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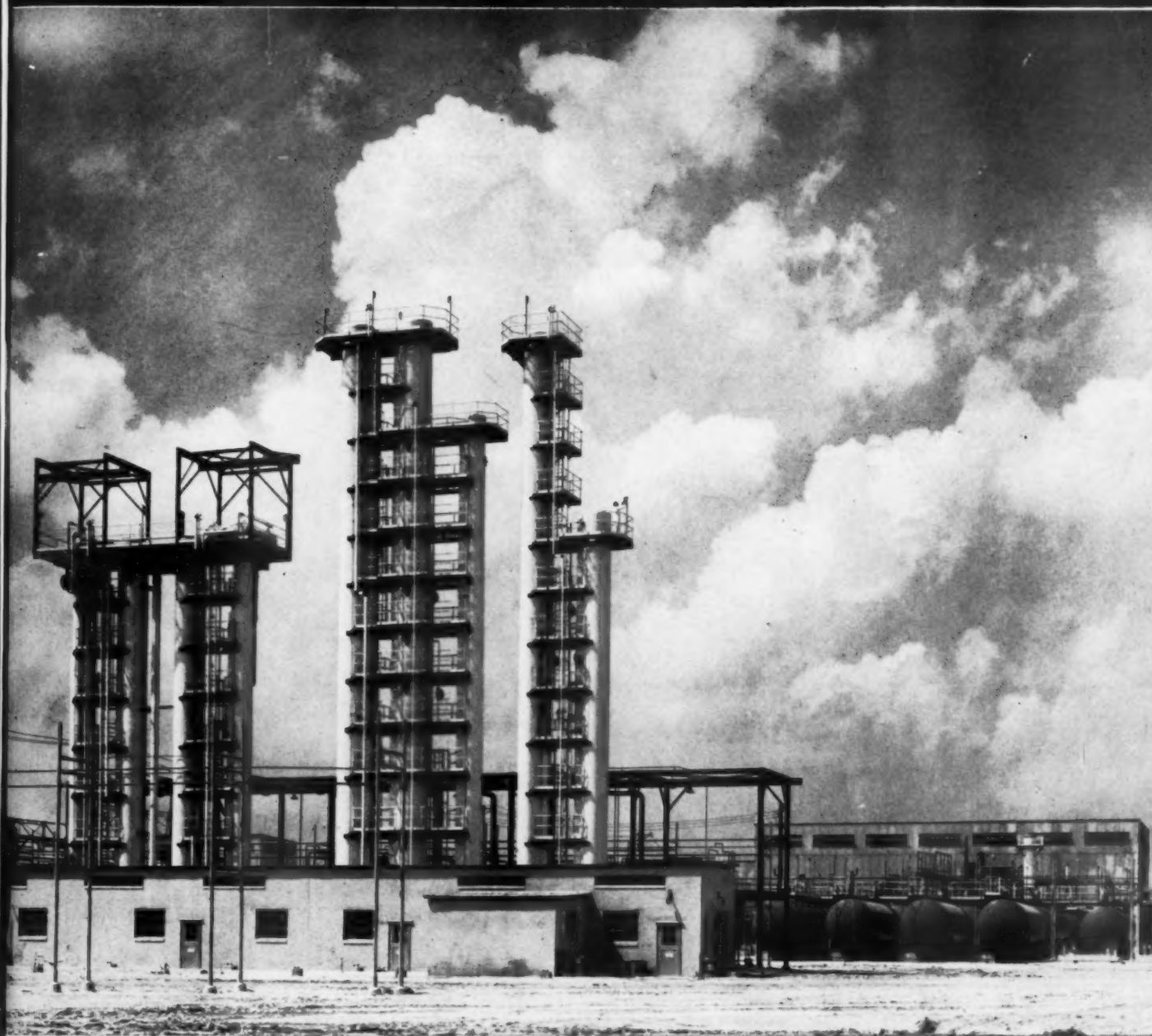
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JANUARY 1960

THE MAGAZINE OF TASTE AND SCENT



Container Development... Page 21 • Phenolic Preservatives... Page 28



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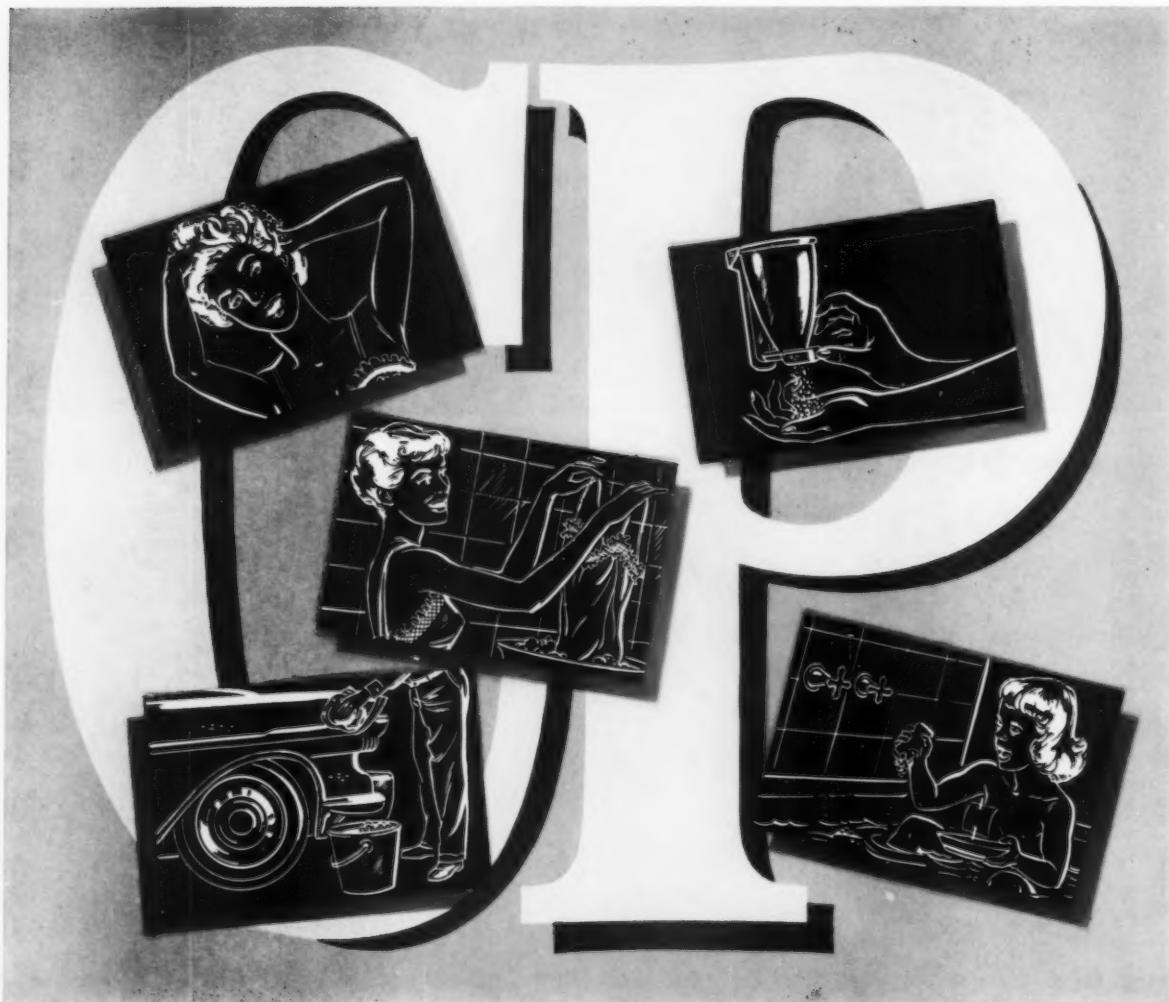
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# American Perfumer AND AROMATICS

VOL. 75, NO. 1

JANUARY, 1960

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## Transition

As we move into the decade that has been optimistically named the Soaring Sixties, we can see major problems looming ahead; problems dealing with the relationship of country to country, company with competitor, man with his fellow man. It is the honesty and strength with which these problems are met that will decide whether this bright new decade we foresee will boom or bust.

In the decade that will undoubtedly project our thoughts further into space, through the courage of the 20th century counterparts of Columbus, Magellan and Hudson, we might find that our perspective is changing. In our haste to gain a greater share of the boom we often find basic values disregarded.

Let us not, in our eagerness to soar, lose sight of those principles by which we learned to stand. For this industry, the Sixties will soar only if we have the honesty and strength and good judgment to make them good years.

We enter this new era with problems that have developed and been carried over; matters of vital concern to the industry and its potential. These problems are not easily resolved. The new era will be a demanding one.

Let us not enter the Sixties with overconfidence, bragging of its portent, then, like our much heralded moon shot of 1959, explain our shortcomings with embarrassment.

We salute the Sixties for what they will be, the years that we will make of them.

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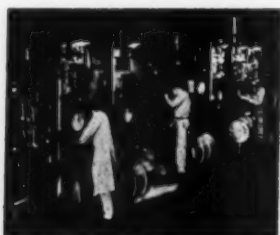
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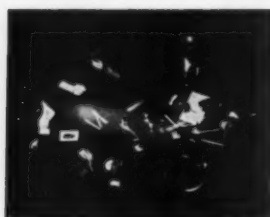
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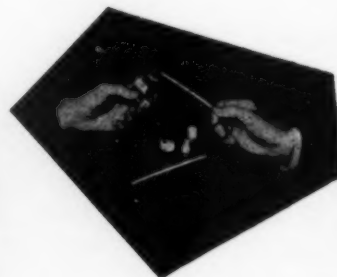
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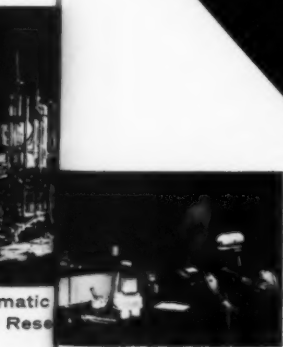
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Oil Production



## Check List of Best Selling Technical Books

- ☐ 1. **COSMETICS, SCIENCE AND TECHNOLOGY.** The editorial board of this encyclopedic cosmetic work is made up of H. D. Goulden, E. G. Klarmann, and Edward Sagarin, executive editor. It is in five parts: I, Scope of Cosmetics (80 pages); II, Toilet Preparations (812 pages containing 800 formulas); III, Manufacture and Technology (214 pages); IV, Physiological Considerations (92 pages); plus preface and complete index. The sixty-five experts who contributed, are a "who's who" of cosmetic chemists in the United States and Canada. 1433 pages. Illustrated. Price \$27.50.
- ☐ 2. **PERFUMES AND THEIR PRODUCTION.** By E. S. Maurer. This is the first text of its kind in the English language to give a complete picture of the perfumer's training, and methods of working. In Part I the reader is offered a complete picture, not only of formulations, but of the entire scope and organization of the perfume industry. Part II is a systematized discussion of all the commonly used perfumery isolates and synthetics. Practical hints on utilization of most of these materials are included, together with notes on their specific applications, blending properties, and other functions. 313 pages. Price \$6.50.
- ☐ 3. **PRESSURIZED PACKAGING (AEROSOLS).** By Alfred Herzka Jack Pickthall. Although technical problems occur with all types of packages, those which arise with pressurized packages are many and complex. This reference book, by two recognized authorities deals with propellants, containers, valves, filling methods, laboratory procedures, emulsified systems, and perfumes. There is a complete section containing more than 200 formulations, including foods, insecticides, cosmetics, paints, and numerous other products. 400 pages. Illustrated. Price \$12.00.
- ☐ 4. **PERFUMERY SYNTHETICS AND ISOLATES.** By Paul A. Bedoukian. This carefully compiled volume meets the genuine need for authoritative data on perfumery synthetics. It embraces the history, chemistry, physical and chemical properties, manufacture, uses, and other pertinent data of the principal perfumery compounds; and covers the important perfumery synthetics. A complete index adds to the value of this important work. 488 pages. Price \$10.25.
- ☐ 5. **HENLEY'S 20TH CENTURY BOOK OF FORMULAS, PROCESSES, TRADE SECRETS—REVISED, ENLARGED EDITION.** Manufacturers, chemists and others call HENLEY'S the most valuable book of its kind. Nearly 10,000 formulas, processes, trade secrets. It contains formulas for nearly everything imaginable; new ways of doing things; technical process; so-called trade secrets. It has helped thousands make more from their present businesses and professions. A single formula may be worth more than 100 times the price of the book. Over 900 pages, completely indexed, cloth binding, \$5.25 postpaid.
- ☐ 6. **COSMETICS—THEIR PRINCIPLES AND PRACTICES.** By Ralph C. Harry, discusses the skin, its nutrition and scientific care: the hair, its proper grooming, the physio-chemical problems involved in its washing; the teeth and their care, covering the present status of different dentrifices, and the luster-producing properties of ingredients. Much of the information has its source in the research activities of its author, embracing chemistry, dermatology, and microbiology. 786 pages. Illustrated. Price \$17.25.
- ☐ 7. **INTERNATIONAL ENCYCLOPEDIA OF COSMETIC MATERIAL TRADE NAMES.** By Maison G. deNavarre, brings you reference data you will turn to constantly . . . the most complete listing of all the materials of the world used in cosmetic manufacture . . . including quick concise descriptions of approximately 4,000 materials . . . the names and addresses of the suppliers . . . and a very useful cross-index of the materials and their uses. You will use it when seeking new materials, or substitutes for those you may now be using. 400 pages. Price \$7.50.
- ☐ 8. **HANDBOOK OF COSMETIC MATERIALS.** (Their Properties, Uses, and Toxic and Dermatologic Actions). By Leon Greenburg and David Lester. Contains alphabetical listing, with frequent cross references, of information on approximately 1,000 substances. For each compound gives: formula (including collateral names), properties, toxic action, dermatological action. Exhaustive bibliography. Essential for manufacturing chemists, cosmetic industry, dermatologists, allergists. 467 pages. Price \$13.50.
- ☐ 9. **THE ESSENTIAL OILS.** By Ernest Guenther, with the collaboration of leading experts, this monumental six-volume work includes: Vol. I, Origin and Development of Essential Oils. 427 pages. Price \$9.50. Vol. II, detailed data on several hundred constituents of essential oils. 852 pages. Price \$13.50. Vol. III, discusses the oils of the plant families Rutaceae and Labiate. 777 pages. Price \$13.50. Vol. IV, covers the widely used oils: citronella, lemongrass, bois de rose, cassia, and others. 752 pages. Price \$13.50. Vol. V, takes up other oils of special interest to flavor, perfume and soap manufacturers: rose, ginger, cardamon, anise. 507 pages. Price \$13.50. Vol. VI, completes the monographs on individual oils: wintergreen, sweet birch, juniper berries, and the numerous pine oils. 552 pages. Price \$13.50.
- ☐ 10. **MANUAL FOR THE ESSENCE INDUSTRY.** By Erich Walter. Comprises modern methods, with formulas for making all kinds of essences for liquors and alcoholic drinks, fruit juices and jams, mineral waters, essences for fruits and other vegetables, essences for confectionery and pastry. Describes raw materials and laboratory practice. Discusses taste and the transfer to foods and beverages. 427 pages. Illustrated. Price \$8.25.

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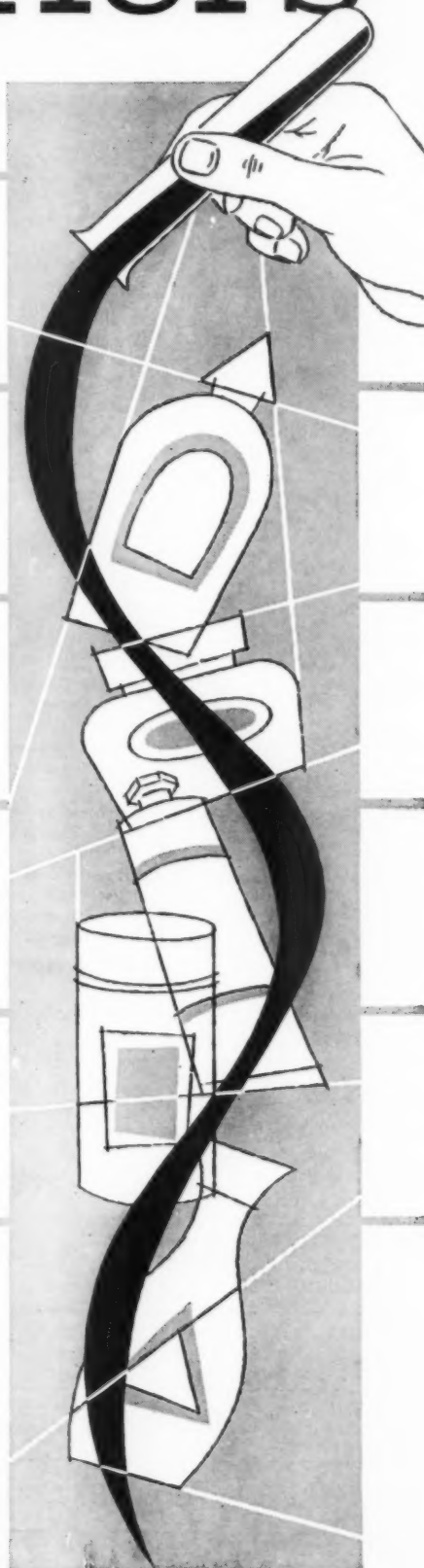
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## MINUTE NEWS . . .

### Food Additives Amendment Time Extension Requests

The Food and Drug Administration has announced its policy on extensions of time for compliance by food and chemical manufacturers with requirements of the Food Additives Amendment. All food additives must be cleared for use before March 6, 1960 unless an extension is granted. Use of a food additive without a formal authorizing regulation or an extension of time after March 6, 1960, will cause the food containing it to be adulterated and illegal for shipment, according to the agency. Requests and supporting data for time extensions should be filed with the Commissioner of Food and Drugs by February 1, 1960. Each request for an extension should give:

- (1) The name and chemical composition of the food additive for which extension is requested.
- (2) A statement of the uses of the food additive for which extension is requested and evidence that these uses were recognized prior to January 1, 1958.
- (3) Information about the physical or technical effect produced by the additive, and the quantity needed to produce such effect.
- (4) Information about the quantity of the additive expected in or to be added to the food.
- (5) Available information which indicates that these amounts of the additive in food will not jeopardize the public health.
- (6) A statement of the reason(s) why a tolerance has not previously been requested.

### Bourjois, Inc. Purchases Helene Pessl, Inc.

Mr. F. X. Patrey, president of Bourjois, Inc., has announced the total acquisition for cash of all outstanding shares of Helene Pessl, Inc., manufacturer and distributor of the "Little Lady" and "Fifteen" lines of toiletries. While HELENE PESSL, INC. will be operated in the future as a wholly owned subsidiary of BOURJOIS, INC., Mr. Arnold Perlman, the founder of the company, will continue in an advisory capacity.

### Effective Date Deferred on Coal-Tar Color Delisting

The Toilet Goods Assn. bulletin #3278 announced the issuance on October 6, which would have delisted seventeen coal-tar colors, the principal use of which was in the manufacture of lipsticks. This order would have gone into effect January 6, 1960. The Food and Drug Administration has deferred the effective date of this order to January 25, 1960, in order that the Commissioner may have more time to study the protests of the TGA and others.

### Favorable Year Forecast For U. S. Fragrance Industry

According to Charles P. Walker, President of International Flavors and Fragrances Inc., 1960 should be especially favorable for the U.S. fragrance industry. "It should," he said, "exceed 1959, when the U.S. sale of all types of consumer products containing fragrances amounted to more than eight billion dollars at retail level." Large scale users of fragrances and fragrance materials, such as the soap, cosmetic, toilet goods and aerosol industries, are expected to operate at a higher level during the coming year, he said.

Especially interesting to the U.S. fragrance manufacturer, Mr. Walker stated, is the continued expansion of the world market for U.S. brand products. This expansion should continue with increased momentum during 1960, particularly in Europe where improved methods of distribution, a marked rise in disposable income, and the lessening of continental tariff barriers should create favorable conditions for consumer products of U.S. producers.

**Pierre Chauvet  
Dead at 52**

Pierre Chauvet, one of the truly great men of perfumery, died December 23, 1959 in Cannes. No tribute seems more timely than the words of his great friend, Dr. Ernest Guenther, in the preface to an article about Pierre Chauvet in *The Perfumery & Essential Oil Record*, March 1954. "I learned to admire Pierre Chauvet: an untiring worker of keen intelligence and enormous energy, a tough and fearless fighter, a man of vision and courage, a warm and devoted friend. A typical son of the Basses-Alpes, his native soil, Pierre Chauvet has already accomplished much more than we ever dared hope in those early days; his pioneering spirit will lead him to ever great achievements. My wholehearted wishes will always accompany him."

**Roure-Dupont Announces  
Indian Factory Opening**

Jacques d'Aigremont, president of Roure-Dupont, Inc., has announced that a new factory has been inaugurated at Sewri by Industrial Perfumes (Private) Ltd. of Bombay. This company was formed in 1957 by the Ets. Roure-Bertrand Fils & Justin Dupont and Tata Oil Mills Co., Ltd. This is part of the policy of Ets. Roure-Bertrand Fils & Justin Dupont to expand their facilities around the world to give better service to their international clientele.

**Phila. College of Pharmacy  
Receives A.E.C. Grant**

The School of Chemistry of the Philadelphia College of Pharmacy and Science has received a grant of \$11,987 from the United States Atomic Energy Commission. These funds are for the purchase of equipment and materials to be used in the educational and training program of that College in the field of nuclear technology. This is the second such grant received by P.C.P.&S. from the A.E.C.

**Robert E. Felton Visits  
Felton Co. (Great Britain)**

Robert E. Felton, vice president of Felton Chemical Co., Inc., manufacturers of perfume and flavor materials, is visiting Felton Company (Great Britain) Ltd. He plans to travel throughout the British Isles with Ray Stansfeld, director, after which he will visit Felton Company-France, to discuss plans for the coming year with Leon Gefen.

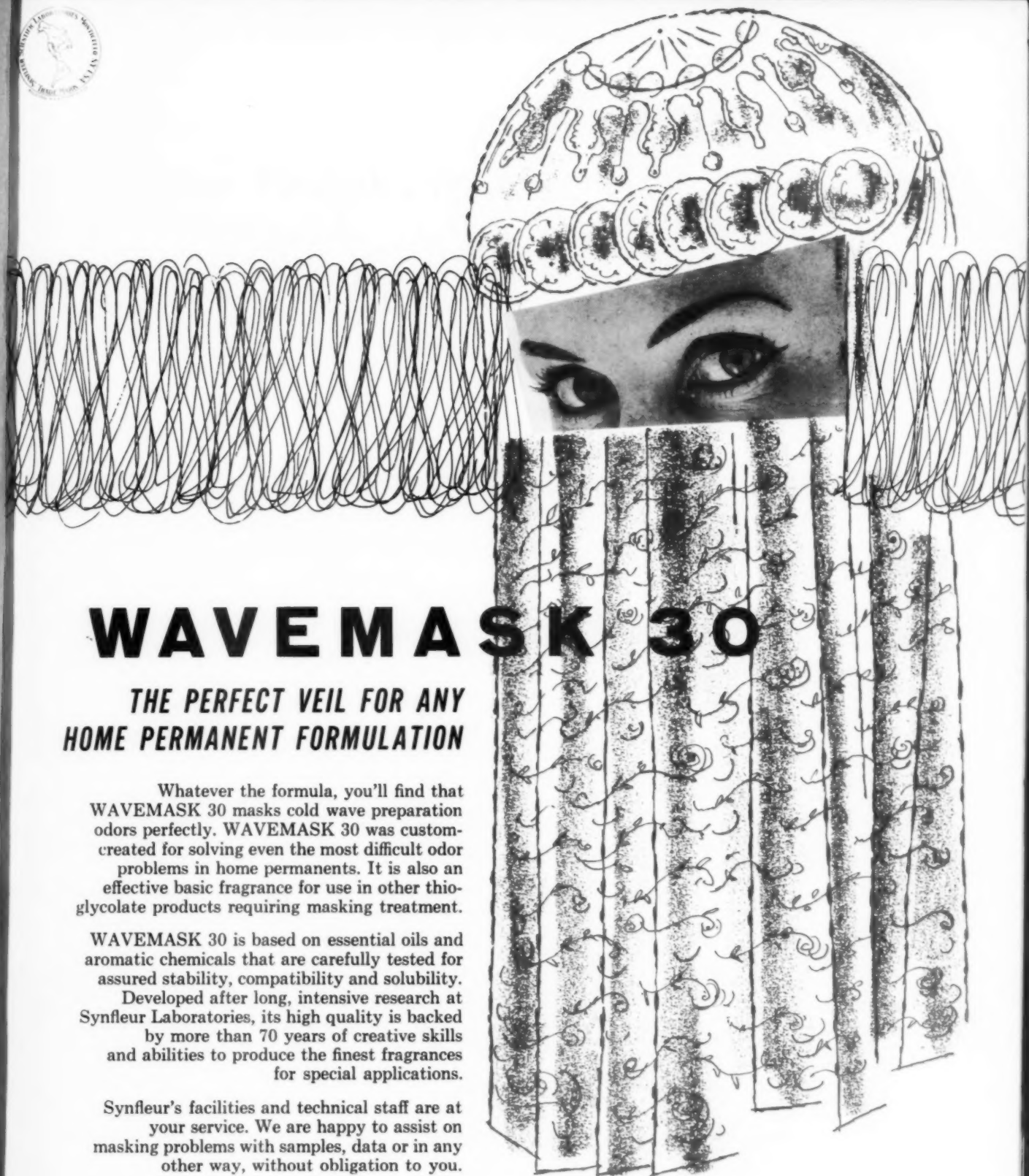
**People**

George E. Best has joined the staff of the Manufacturing Chemists' Assn. as staff secretary to MCA's committees on air and water pollution abatement and chemical packaging as well as to handle special assignments. Dr. Joseph V. Swintosky has been appointed Principal Research Pharmacist of Smith Kline & French Laboratories. Paul Byrne has been appointed New Products Manager of Colgate-Palmolive Company's Toilet Articles Division. Wesley E. Gatewood has been named director of field sales for the Barrett Division of Allied Chemical Corp. Thomas S. Moroney has been appointed as a sales representative for the Cleveland District of the Silicone Products Dept. of the General Electric Co. John R. Miller has been promoted to Eastern Sales Manager of Florasynth Laboratories, Inc. Dr. Robert W. Cairns, director of research for Hercules Powder Co., has been elected a member of the board of directors. Roderic L. O'Connor, a former Assistant Secretary of State, has been elected to the Boards of Director of Ciba States Ltd. and of Ciba Pharmaceutical Products, Inc.

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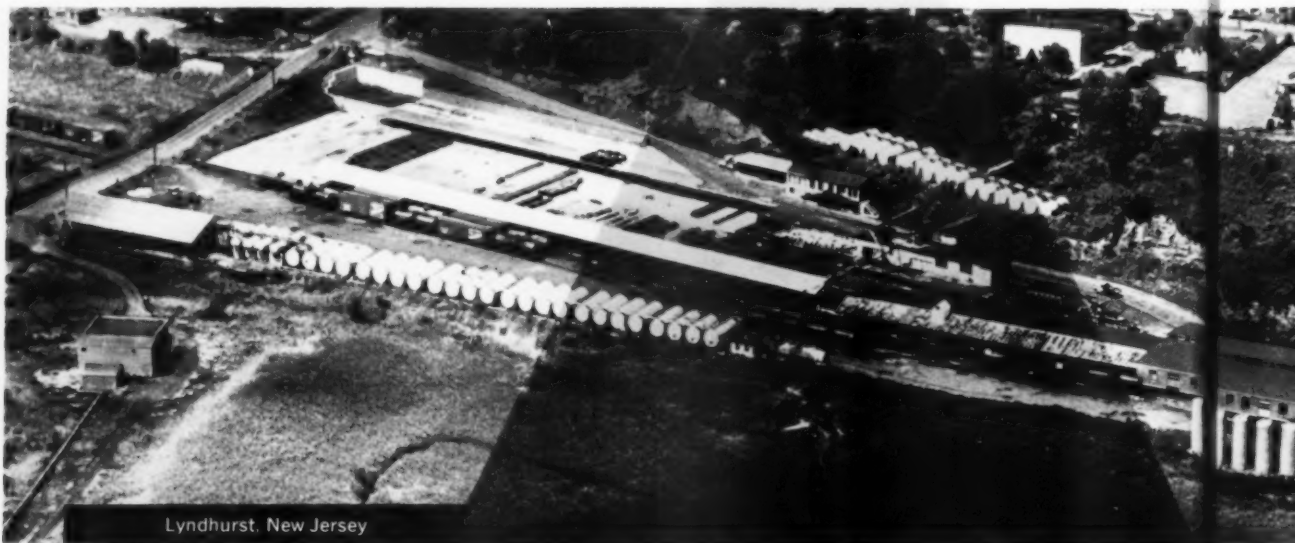
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THIS WAS HUISKING IN 1910...

# 50<sup>th</sup> Anniversary Report:

THIS IS HUISKING IN 1960



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Today, Huisking remains a principal factor in import, export, and distribution of pharmaceutical and industrial chemicals. Its manufacturing divisions are steadily increasing operations. Glyco Chemicals division manufactures chemicals for the needs of the food, cosmetic, plastics, paper, electronics, metals, paint and textile industries, expanding its product lists constantly. Peder Devold Oil Company division is a primary source of Cod Liver Oil, Feeding Oils and other Vitamin Oils. Clintbrook Chemical Company division produces Pharmaceutical Chemicals.

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# Q UESTIONS & A NSWERS

## 1361: LUMINOUS LIPSTICK

**Q.** Please send us a formula for making white lipstick of the type now being sold—so-called luminous. Can you tell us who supplies Xerol "T." D. M., Mich.

**A.** In making the white lipstick, use white pigment such as titanium or zinc oxide or both from 5 per cent up in a regular lipstick base using the usual procedure for manufacture. The R. W. Greeff and Co., Inc., 10 Rockefeller Plaza, New York 20, New York, supply Xerol "T."

## 1362: HAIR PREPARATIONS

**Q.** Would you please give us a reference regarding the formulation of hair preparations for both the white and colored races? C. T., Virginia

**A.** We are unable to give you any references regarding the information of hair preparations for white and colored people other than such data that appears in a standard text on cosmetics. There are several of these as you undoubtedly know and the most recent one being that by Sagarin, COSMETICS: SCIENCE AND TECHNOLOGY. If any book would contain such information, this one would.

## 1363: SOLUBILIZER

**Q.** We manufacture a pine tar shampoo using a 6040 cocoa base, but we have difficulty in getting the pine tar oil to dissolve completely. Could you tell us something to add to this solution to dissolve the pine tar oil? We will appreciate this information as quickly as possible for which we are enclosing a self-addressed, stamped envelope. L. C., Utah

**A.** You should try one of the solubilizers, such as one of the Tweens, G-2160 or related materials, all offered by the Atlas Powder Company, Wilmington 99, Delaware, for the purpose of dissolving your pine tar in the shampoo base. We suggest you write to Atlas Powder Company, telling them your problem. We are sure they will be able to suggest a proper solubilizer.

## 1364: INFORMATION

**Q.** At the present time we are in the process of constructing a booklet on information pertaining to perfume. Would you be so kind as to forward information such as the manufacturing process, ingredients used and anything of interest that might be available. Any effort on your part would be greatly appreciated. E. A., Mass.

**A.** Our best suggestion to you is to go to the public library and read any of the several booklets on this subject. I am sure your library has many of these books. There are too many to mention here, but you could refer to COLA Cerebalaud books as well as to the works of Poucher.

From time to time suggestions have been and will be made in this magazine with respect to processes, devices, materials, appliances, equipment and the like. It is not practicable for the writers and editors to have a patent search or examination made in connection with each such suggestion. Our readers are, therefore, requested and indeed urged to determine for themselves whether any patent or other right will be violated before acting on any such suggestion.

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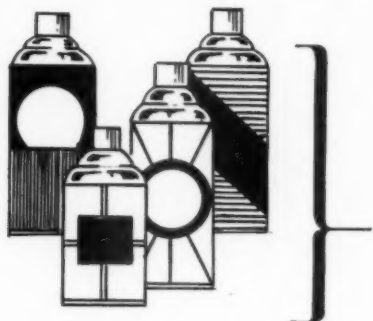
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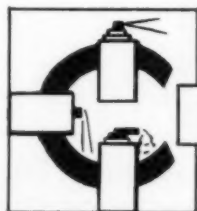


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## DESIDERATA

Maison G. deNavarre, M.S., F.A.I.C.



### NEW TRANQUILIZER?

All the stuff used by witch doctors and native folklore cannot be brushed off as so much baloney. Rauwolfia again proved it to us.

Now it is a shrub member of the pepper family used in Polynesia under the names of Kawa, Kava or Ava. The root has been highly regarded as producing a potion that reduced "fatigue and produced complete freedom from anxiety" according to a paper by Klohs, Keller, Williams, Toekes and Cronheim in J. MED. PHARM. CHEM., 1, 95, 1959. One senses it took five men in shifts to do the ground work due to the activity of the tranquilizer. The authors have a fraction from which they isolated a compound A ( $C_{14}H_{12}O_3$ ). This is responsible for its activity.

### PROLONGED USE OF ESTROGEN

The November 21st issue of J. A. M. A. has an interesting article on the prolonged use of estrogens (diethyl stilbestrol and conjugated equine estrogens). Among the observations found in 292 women in which the mean duration of therapy was 5.1 years, there was "no justification for the fear that mammary and cervical carcinoma may result from this therapy" according to Wallach and Henneman. This was actually a 25 year retrospective study of the results of prolonged use of estrogens by humans.

More such studies would be desirable to clarify the use of estrogens internally and externally over extended time periods.

### NEW CELLULOSE ETHER

A new supplier of hydroxyethyl cellulose offers the material in three viscosity grades, in the form of a free flowing granular material readily soluble in water. It is nonionic. Acids and alkalies have little effect on viscosity. It has improved tolerance for lower aliphatic alcohols. It is compatible with natural and other synthetic gums. It finds use as a thickener, protective colloid and binder.

### NOTES

Our compliments to Harry Hilfer to whom U. S. Patent No. 2,890,987 has just been issued. Covers astringent compounds in stick form. . . . Wonder how crow tastes without cranberries? . . . Don't know what this will do to some people's ego not in the U. S. A., but a world poll made the French Institute of Public Opinion say the U. S. has the highest culture in the world. . . . Desperoids finds that royal jelly produced by bees may vary in composition considerably depending on the source of its food. Thus, bees fed on pollen of blossoms of chestnut trees have a much higher nicotinic acid content. . . . Babicka who has studied the microbial flora of packaging again writes about the contamination due to closures with cardboard or cork backing used with a protein containing adhesive. . . . Iodine baths are said to have a "trophotropic and stimulating effect" and should be used in the form of elemental iodine for best results. . . . One German company is offer-

ing an aluminum gauze dressing for wounds and burns. . . . While new chamomile hybrids produce larger blossoms with a higher active ingredient content, the petals are more fragile, breaking off in handling according to Chladek and Kosova. . . . As we go to press, there is an announcement of a paper by Kauzmann to be given before the section of Chemical Sciences of the New York Academy of Sciences on December 1st, regarding the increase in volume of proteins when in contact with acids, bases and urea. Hope you see a possible relationship between certain cosmetics and skin. . . . A mixture of bentonite, cetyl alcohol, yellow beeswax and mineral oil can hold 50 per cent of water. . . . Coconut diethylamide will dissolve lanolin and render water dispersions of the two clear and transparent. . . . Have spent a few days with "Reggie" Warner (John Gosnell and Sons, Lewes, Sussex) who was here on business. Brother Leslie Warner (Courtin and Warner) was the one who put me straight on Bisquit Dubouche. I introduced Reggie to Crown Royal and Melcher's Aristocrat. We both have been testing the Crepes Suzette at the Maison Riviera (Detroit) and Town and Country (Toronto). . . . Got a nice compliment from Ed Breck via long distance on the last issue of the AMERICAN PERFUMER. . . . The more I think about it, the more I wish Frank Chilson would change the name of his invention from Chilsonater. It sounds too much like something else. . . . Had a real thrill at the press show-



## ● EMULSIFIERS

## ● DETERGENTS

## ● ABSORPTION BASES

## ● FRAGRANCES

### ● SCIENTIFICALLY DEVELOPED

AND MANUFACTURED—

SPECIFICALLY FOR APPLICATION

IN COSMETIC FORMULATIONS



ing of Toni's newly expanded labs. Missed Joe Kalish but did see lovely Veronica Conley. Ray Reed hosted at lunch with a short talk afterward. It is quite a layout with over 200 technical people on two floors of the Merchandise Mart. Will do more on this in another issue. . . . Users tell me there is a problem with this stuff you paint on the skin and in a couple of hours you have a Florida tan. You miss some spots and they stay white. Where you double up by overlapping application you are darker. . . . Guess Freddy Wells and all the boys in England are in arms because a certain employment counselor has solicited one and all for a highly paid job in the cosmetic industry. There was some comment on it when I was there in September. Freddy, it's being done all the time over here. Just got an offer myself for a similar deal. They run about one a year. I'm still at the same old stand and like it. Hope to be there until Social Security . . . but am changing secretaries—it seems they want to get married. Much happiness to you both, Patty. So, dear reader if your correspondence is a bit delayed, you will know why. . . . Welcome Home, Bob Kramer. You will have to tell us all about your around the world hop. Got your cards so I know where you were—but now want the low-down. . . . Necessity is the mother of invention. Have you tried the new resin for hair sprays offered by National Starch? . . . Antonin Zenisek certainly keeps me up to date on all new stamp issues of Czechoslovakia. His paper on urocanic acid which we recently published has raised some interest in the material. Unfortunately, it is not commercially available, to my knowledge. . . . Geigy have a new germicidal agent, a chlorinated salicylamilide. . . . Glycerine Producers Association have hit on quite a novel idea for promoting use of glycerin cosmetics. You'll be seeing it soon.

**ENCYCLOPEDIA OF CHEMICAL REACTIONS, Vol. VIII, by C. A. Jacobson and C. A. Hampel. Reinhold Publishing Corp., New York 22, N. Y. 1959. 533 pages, indexed. Price \$14.00.**

This ambitious series is complete with the present volume which covers tungsten through zirconium. The material is alphabetically arranged as to reacting substances and reagents. All known reactions are included. In addition there are 768 references on elements discussed in previous volumes.—M. G. deN.



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Blue marble jars for cosmetics, Egyptian, 2000-1800 B.C.

## Developments in Rigid and Semi-Rigid Containers



JOHN M. SHARF, *General Manager*  
*Packaging Materials Research*  
*Armstrong Cork Company*

THE RESEARCH AND DEVELOPMENT worker is faced with a dilemma of evaluation when attempting to utilize the ever increasing variety of rigid and semi-rigid materials in fabricating containers for the present day variety of foods, drugs, cosmetics, chemicals proprietary products, beverages, etc. in which occur solids, liquids and occasionally gases. The interpretation as to what may be allowable package function or tolerable characteristics of the material is invariably keyed to proposed shelf life and potential change of the contained product in keeping with the impressions of sales and merchandising demands, not overlooking at any time the economic cost factors of the package, its processing, transportation and utility.

A definition or guide post is well warranted; namely, "The ideal container or packaging shall maintain the contained product in the original condition as prepared by the manufacturer, neither interacting with nor contributing to the contents; it shall adequately protect against potential changes likely to be induced by external influences during processing, storage, transportation and handling to the point of ultimate consumption."

Note that the definition does not mention time as

This paper was originally presented in its entirety at the 21st Annual National Packaging Forum of the Packaging Institute, November, 1959. It was selected for the TOC Award which is given to that individual who contributes the most informative paper at the Annual Forum of the Institute.

such, the imponderable to be determined by the processor for each item. Historically speaking, rigid containers have endured and functioned for centuries. The amphorae of glass or pottery containing oil, wine or beer recovered from archaeological sites or from ancient submerged shipwrecks have been of recent news interest, particularly since the products survived without appreciable alteration. Likewise, containers of noble metals or bronze and copper from these ancient sites have preserved many unguents or powders for the review of present generations.

In each instance, the rigid container survived the various forms of natural violence and external influences surrounding it, keeping the product in remarkably good condition.

The fore-runner concept of the modern rigid container is somewhat over a century old. Appert prepared some of the first preserved foods in glass containers during a several year period up to 1810, at which time he published his findings and won a prize from Napoleon (1). Shortly thereafter, the metal or tinned cannister was produced by Durand in England for similar usage. The application and widespread usage of these two types of rigid containers has been a great accomplishment in control of raw materials, fabrication, use of highly automated production machines and the establishment of closely controlled uniformity, all at very reasonable economic prices. With such containers numerous types of packaging industries have prospered.

Obviously glass containers, the modern version of the amphorae, have become more utilitarian, lower in cost and widely employed because of maximum inertness. Metal containers have departed from the noble metals, bronze or copper to the more economically feasible tinned steel or aluminum plate, in most instances protected with specialized interior coatings to extend service life against aggressive products.

The present era of semi-rigid containers is relatively recent and is based upon the variety of man-made polymers, principally thermoplastic. The primary cost of the materials favors formation in thin sections to meet economic competition, and in some instances, the resulting flexibility may become an inherent feature of the container. Fabrication methods and machines have been developed to meet the specific characteristics of each, for it is not always feasible to have "universal" fabrication equipment to inject, form or blow the materials interchangeably, particularly if dimensional control is critical. The complexity of the problem becomes apparent upon the realization that one general classification recognizes fifteen or more classes of these thermoplastic raw materials, each with definite and sometimes divergent problematical physical characteristics.

It is, in fact, the variety of physical performance characteristics which has led to difficulties in commercial evaluation. This is particularly true since no one of the semi-rigid materials will entirely fill the requirements of an ideal packaging material. As a result, the final package must often be recog-

nized as one of limited shelf life, since there can be product changes with time or the container itself may show evidence of undesirable change.

Therefore, it is advisable to review each of the major evaluation factors that must be applied to both rigid and semi-rigid containers to assure commercial performance. These evaluations, for the greater part, require laboratory investigation under exact or simulated life cycle. The data that result should be carefully interpreted by competent technologists. Unfortunately, the development era of these polymers and the resulting containers has led to a variety of test methods, quite often reported in publications, but the data given are not always useful for direct comparisons or to evaluate products of differing composition.

**1. Mechanical Strength**—The container should, when called upon, have sufficient rigidity to protect the contents if they are of discreet shape or size, so that in storage or transportation the product is not broken up or the container distorted. Good mechanical strength results from cylindrical to oval shapes, of heights not more than several diameters. Further, certain mechanical rigidity is necessary to go through modern packaging lines having indexing mechanism on conveyor lines, holding chairs on fillers and particularly under the clamping mechanism accompanying the typical capper or closure applying machines. The conventional screw cap or other closure requires torque strength of container necks and openings, or the closure may have to be re-designed. If the container is to carry pressure above or below that of the surrounding atmosphere, the material should have adequate structural rigidity to prevent distortion or collapse. This problem is found with the changes in barometric pressure that accompany changes in elevation, say from the sea coast to Denver, as well as changes of internal pressure which often accompany product aging or temperature changes. Another associated factor to be commented on later concerns the apparent changes in the mechanical strength of some materials when in contact with the product for any unusual length of time causing softening and thereby slumping. With modern multiple tier palletizing, the container must show sufficient strength to withstand the rigors of shipping without undue reinforcement of box or separator arrangement. With all materials, it is important that they do not change shape, but show dimensional stability upon aging to be certain of mechanical match with component parts and assure proper functions.

**2. Resistance to Contents**—Obviously the contents should not dissolve or corrode the container materials. This factor is difficult to qualify under all circumstances because the potential range of products to be packaged may vary from strongly acid to strongly alkaline, may be oily substances, detergents, materials of low surface tension, a wide variety of organic chemical solvents, etc. In this function, glass containers are recognized by the United States Pharmacopoeia (5) as being most universally resistant to a full range of products,



even under extended periods of storage. Such resistance to a wide spectrum of products is not matched by other materials, each of which must be qualified. Studies originating a number of years ago and extended by various later experimenters indicate that there are definite limitations in containing a wide variety of commercial preparations on the basis of plastic resistance to the contents (6). A more subtle problem of resistance of the plastic materials to contents will be covered later under the topic of environmental stress cracking.

**3. Thermal Resistance**—Wherever products are given any sort of processing in the container to assure stability or to control micro-organisms through pasteurization or sterilization, the container must withstand the maximum temperatures and time encountered without distortion or breakdown. Conversely, if heating is necessary to prepare the product for dispensing, the same reservations apply. Many of the thermoplastic materials have relatively low heat distortion points and, therefore, are limited to this type of package function. Prevailing temperatures may also influence mechanical strength, for some plastic materials on being cooled to refrigerator temperatures or lower become somewhat "cold short" and will fail under relatively light load, particularly if there is some impact involved. While low temperature performance may not be necessary in ordinary service such as dispensing the contents, yet there is very chance that the container may be involved with low temperatures during winter shipment. Obviously, such materials should not be employed in containers intended for low temperature service.

**4. Water Vapor Transfer**—Water vapor transfer through the container wall may be sufficiently high, allowing moisture to migrate away from the contained liquid product causing the lowering of the fill line, changing the consistency or efficacy of the products. On the other hand, the reverse may be true if moisture can, under conditions of high humidity storage, migrate back into the product and, in case it is a dry product, change its stability or finished texture. In the instance of many drug products of specific strength, the migration of moisture into or from the product through the container can be serious. If the water vapor rate is relatively higher than a return transfer rate of oxygen and nitrogen of the air, the headspace pressure will drop and the container walls are likely to buckle. Laboratory evaluation will make possible an estimate of the volume percentage loss that may be expected over a typical year's storage life, and a nominal pass level is not over 3% per year, with some discrimination necessary. Unfortunately, small containers have a high surface to contained volume and they show disproportionate fill level change with such loss. Further appreciable loss causes divergence from label content declaration, a matter of regulatory concern.

**5. Gas Transfer**—This phenomenon is somewhat related to the migration of water vapor through

the walls of the container, but may be of different magnitude, quite unpredictable from the knowledge of the water vapor transfer rate. The most serious gas to be considered is oxygen, for many products are oxygen-sensitive and will change in color, taste or active ingredients upon a gain in oxygen. On the other hand, it is equally as important that there not be outward migration of gases, such as carbon dioxide or nitrogen, from the container in the event that such gases are used for inert atmosphere to control quality. The same viewpoint will apply to other purposefully added gases. These transfer rates can only be determined by actual measurement or by long time storage tests of products under representative circumstances.

**6. Permeation of Product Components**—The permeation or drenching of certain product components into the physical structure of the plastic can become complex. In some instances, it is possible for various ingredients to migrate through the wall and appear on the outside as a greasy film or as a crystallized substance. This migration has also been of concern with some plastics when active ingredients disappear into the plastic structure out of the product or controlled dosage. In a similar manner, odorants, flavoring or perfuming agents can disappear from the contents, changing the overall character of the product or causing it to be constantly variable. Adjustment of the formulation may be a partial solution to extent the nominal length of storage or service life. This permeation phenomenon is often accompanied by a softening of the plastic materials and, as such, may contribute to bulging, slumping, loss of shape and, if the exterior surface changes, to detachment of labels.

**7. Environmental Stress Cracking**—Related to the problem of drenching or permeation some plastics show a tendency to craze or crack, particularly when loaded or stressed during contact with a product as found with screw closures or during flexing in application. This environmental stress cracking evaluation requires exact test procedures before it can be fully evaluated, since it is the combination of the migration of the product into and disruption of the molecular structure as well as mechanical loading. A specific test detail has been recognized for nearly ten years as the Bell Laboratories Test (2), or later versions (3). However, actual tests should be tried with specific products in the exact containers proposed under simulated cyclic conditions of usage, particularly as to mechanical loading, if the container involves multiple service dispensing.

**8. Resistance to Micro-Organisms**—In some instances, there have been indications as to the potential growth of micro-organisms, such as molds, either on or associated with the container material, due to migration or concentration phenomena. These effects can be unsightly and may attract insects or other warehouse pests. When transfer rates are high, nutritive materials may possibly

(Continued on page 54)



# Kolar LaboratoriesM

**50 YEARS AGO** a young pharmaceutical chemist established the first exclusively private brand cosmetic manufacturing business. Gustav S. Kolar's boyhood interest in the magic of chemistry had come full circle. A graduate of Northwestern University in 1903 with Ph.G., and in 1904 a Ph.C., his first exposure to commerce was with the pharmaceutical firm of Stearns & White, a private brand manufacturer. In 1905, he studied with Prof. Julius Stieglitz at the University of Chicago, and in 1906 was offered the Civil Service position of Chief Chemist of the City of Chicago. He declined this in order to use his recently acquired knowledge to create a scientific approach for the embryonic cosmetic industry.

G. S. Kolar joined forces with Mme. QuiVive, a nom-de-plume for Mrs. Helen Jameson, the King syndicated beauty editor. Acting as her Chief Chemist in the development of the Mme. QuiVive line, many practical innovations were immediately introduced. At that time, steam distillation of flower petals was a prime source for essential oils in Europe. The oil was skimmed off of the distillate and the remaining waters such as rose, orange, elderflower, etc., exported to the United States in demijohns. Here these waters were utilized as the aqueous phase of the creams and ointments which were manufactured here.

As one would expect, these creams and ointments developed a non-appealing odor in a short time. Kolar's first work was to purchase the pure oil, and with the aid of distilled water and the food approved preservative, sodium benzoate, manufactured a stable product.

Also, during this period, he purchased imported

Russian mineral oil as a substitute for the commonly used imported almond and peach kernel oil for use in the manufacture of a stable, appealing cream. Rancidity of the oil was thus circumvented, and by using beeswax and borax, the forerunner of present day cleansing creams was achieved.

The first hair dye in the QuiVive line was manufactured of silver nitrate. This product was received by the public and even now formulas of this type can be purchased on the market.

In 1910 Kolar Laboratories was officially founded and the creation of new products was demanded by

Kolar's Chemical Laboratory. Peter Parker, right, chief chemist, is making microscopic examination while other members of the laboratory are making weighings or running titrations. This laboratory is used for quality control work of finished and raw materials as well as product developmental work.



This group represents 169 years of cosmetic experience. Left to right: George G. Kolar, president and son of founder; Hy Wasserman, who joined the company 46 years ago and has devoted himself exclusively to sales; Gustav S. Kolar, Founder and Chairman of the Board; Mr. H. R. Kolar, brother of the founder and chemist with the organization since 1912.



## sMarks 50th Year

an awakening consumer public. A new development of that year was the manufacture of an improved nail polish. At that date pastes or powders of tin oxide and pumice stone were the chief buffing agents. It was discovered that the silicas, amongst them infusorial earth, could be compressed in cakes for easier use. Then an alcoholic suspension was achieved with the introduction of a staining dye. Thus was the first tinting nail polish produced. The dye matter used at that date was cosine.

In 1911, G. S. Kolar employed his brother, Hugo R. Kolar, later to become a partner, and to this

day a member of the nucleus of the Company's product development group. His vast background is of great value to the newer men in the organization. The expanding business then moved in 1913 to what proved to be a rather permanent location at Seeley and Madison St. in Chicago. At this time, liquid creams became quite the vogue. This trend has now completed a cycle and we are experiencing a revival today. At that time, stability of emulsion was due in part to the fine peach kernel and almond oils which were obtainable.

A new member of the organization, Hy Wasserman, gave sales an impetus soon after joining the Company in 1914. The laboratory then entered into the manufacture of pressed compact rouge involving the use of punch presses. Lipstick molding operations were put into production. However, the major sales increase resulted from the solidification of the conventional hair oil. By combining ozokerite and paraffin with a mineral oil base, a "concrete" form was produced. This type of product reached its greatest sales during the period of Rudolph Valentino's public idolatry.

The War years brought many obstacles to all chemical based industries. Lanolin, which even then was considered a necessity to all good "skin food" and "nourishing" creams, was cut off. The Kolar's at the outset descended on the wool washers in Chicago and did their best to assure an adequate supply of raw material which they refined.

After the Armistice, and with the beginning of the "flapper era," new demands for cosmetics were created. Kolar's greatest success of this period resulted from the introduction of a waterproof cream rouge which would change color upon appli-

A portion of the liquid filling lines. Materials are fed to the lines palletized and then fed to the filling machines from unscrambler tables. Depicted here are vacuum filling machines. Due to the need for flexibility in private label business, note that this filling machine can feed into three different capping lines.





cation to the skin. This was the first nationally advertised item of its kind, and received the benefit of Charles Walgreen's aggressive merchandising. Products developed by Kolar for its clients in the early 20's were; a marcelle wax in bar form, consisting of carnauba and assorted waxes which allowed the consumer to run the curling iron more easily through the hair; a rosin type depilatory; the use of perborates and peroxides for bleaches.

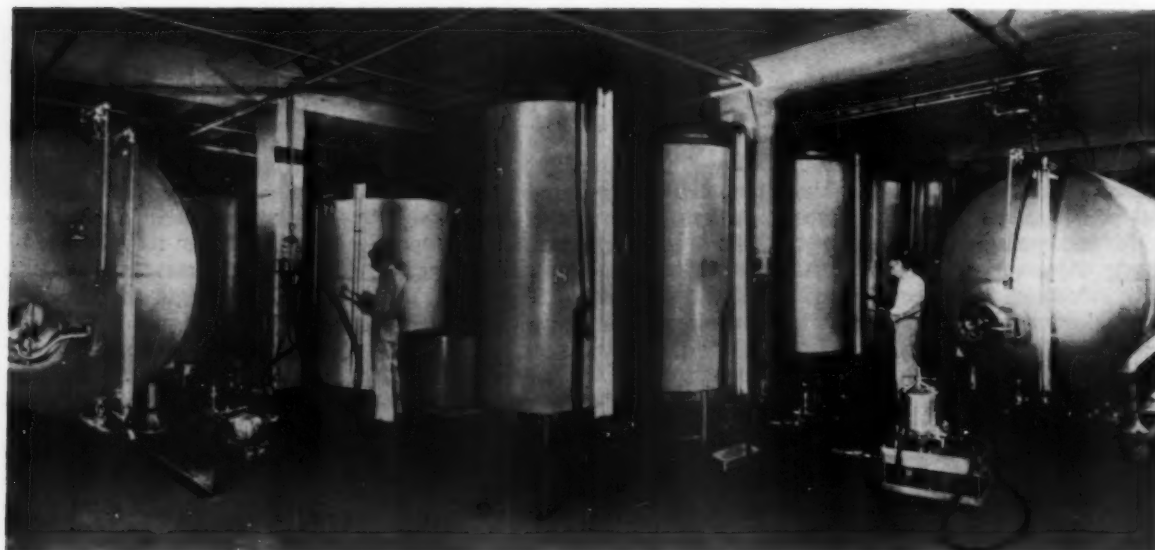
Nessler's invention of the permanent wave machine created a great demand for products to aid in the processing of hair. One of Kolar's products of the era in this field consisted of the manufacture and impregnating of wool pads with a sulfonated oil solution. Thus, a softening action on the hair was achieved and the devastating effects of borax solution were minimized.

Sulfonated oils, primarily castor, were used in the very early 20's for their cleansing ability. Advertisements proving that oil and water were miscible astounded the public.

One of the first home permanent wave kits was



Left to right: Lester Bartolai, purchasing agent, an employee for 38 years; Chester E. O'Sheil, vice president and general manager, an employee since 1940; Edwin Michalik, assistant treasurer, an employee since 1949; Charles Ohanian, traffic manager, with Kolar since 1946.



introduced in the late 20's and sold to the leading mail order houses. This consisted of two vials of chemicals; one, the waving agent, being sodium sulfite, and the setting chemical; the other vial, citric acid. These were diluted by the consumer and applied to the hair. A number of metal hair clamps with a hot plate were the remaining items of the kit.

Hair products in the late 20's and 30's received great attention. Hair conditioning solutions manufactured of keratin and cystine proved to be a boon to the hairdresser. The introduction of bactericides and germicides into hair grooming products already then were widely advertised and demanded by the public.

In 1938 the second generation was introduced to the business. G. S. Kolar's son, George, was

graduated from the University of Chicago in Chemistry and immediately went to work in the laboratory. A knowledge of the other phases of business had been acquired during previous summer work. Expansion of the organization during this period resulted in the appointment of Peter Parker as Chief Chemist in 1940, and Chester E. O'Sheil as Production Manager in 1940.

Intensification in the research of hair and skin chemistry followed. The industry was concentrating on the "cold" waving of hair. Kolar Laboratories, after amassing a wealth of toxicological and pharmacological data on thioglycolates, introduced to the public the first home cold permanent wave kit in early 1942.

Despite the absence of key personnel and ability to obtain prime materials during the war years,



Kolar's growth remained steady. Thanks for this go to a nucleus of seasoned personnel under G. S. Kolar as President, Lester Bartolai as Production Manager, Joseph Kieffer, in charge of Manufacturing, Miss Bee Schuerr in charge of Office Operations, and H. R. Kolar in the laboratory.

Concentration in the development laboratory on protective creams for the various new problems arising from the employment of women in the burgeoning war industries further expanded the scope and volume of the Company.

Upon the conclusion of hostilities, George G. Kolar assumed the Presidency of the Company in 1946, and with the return to the Company of the men in service, a drive ensued to utilize the new procedures and equipment available. The wartime experiences with the new methods of materials handling and packaging equipment caused a metamorphosis in production.

The laboratory applied the accumulated knowledge in the home permanent wave field to the development of the first commercially successful

quate and caused Kolar to purchase buildings of 138,000 sq. ft. in late 1957. This plant is centrally located, one mile from the downtown Chicago Loop, and with easy access to all trucking lines.

This steel and concrete fireproof building has permitted the installation of the most modern manufacturing, packaging, and warehousing equipment. Continued concentration on the development of new products and Kolar's central midwest location has been conducive to their constant growth.

Due to the confidential nature of private brand work, it is not possible to publish the names of the nationally advertised products or the concerns for whom Kolar manufacture. However, many recognized distributors have found Kolar's services always rewarding in the manufacture of specialty items or entire lines.

Packaging lines now vary from semi-automatic equipment for short run specialties to lines of rated speeds of 60, 120, and 240 units per minute.

The organization, due to its stability and age, has many employees of tenure and experience. ■

◀ A portion of Kolar Laboratories cold mixing, manufacturing, processing, and storage tanks. Large stainless steel tanks are 3,000 gallon capacity with propeller type agitators. These tanks are used for alcoholic and oil products.

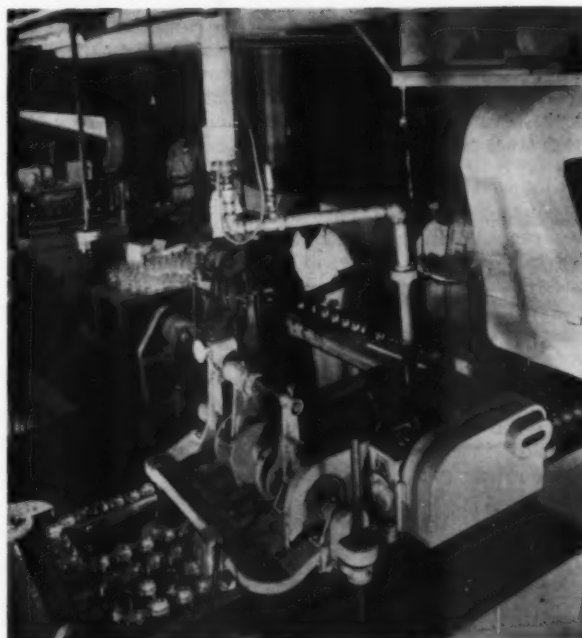
Fully automatic liquid filling line shows 20-spout vacuum filler. ▶ Bottles come from unscrambler table to the left and are conveyORIZED to the filling machine, which then carries the bottles thru the Automatic Capper, Labeller, and Automatic Cartoner.

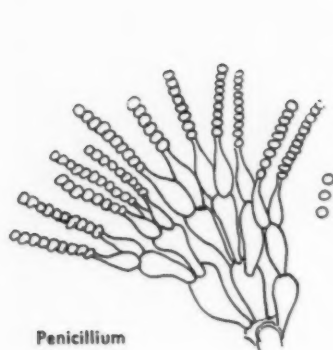
One of the fully automatic quadruple piston filling machines. Creams or lotions are pumped from manufacturing departments thru insulated pipes into machine filling manifold. The jars are filled 4 at a time at a speed of 120 jars per minute. After the cream is cooled in the refrigerated tunnel, the jars run through the automatic capping machine in the background. ▶

thioglycolate hair straightener. Kolar approached the study of antiperspirants from a new angle. Pioneering with others, the practicability of buffered aluminum salts and a greater investigation of the role of skin bacteria in the development of body odors resulted in the manufacture of more effective and longer lasting deodorants.

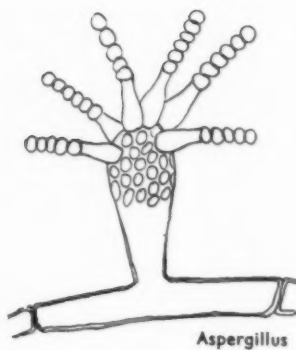
Long experience in the study of hair and skin pigmentation from early work with hair coloring products resulted in grants to the University of Michigan to advance study of the D O P A mechanism. Work also at the University of Michigan in 1946 under the direction of Dr. Carey P. McCord, produced the first known use of radio-active tracers in the study of cosmetic products.

After 39 years of growth at the Seeley Ave. address, a fire in 1949 caused the sale of the property and a relocation at the Rogers Ave. plant. With the continuous development of new products in the laboratory and an ever-expanding group of clients who nationally advertise proved this plant inade-





Penicillium



Aspergillus

## Some Physico-Chemical Aspects of Phenolic Preservatives in the Presence of Macromolecules

H. B. KOSTENBAUDER, Ph.D.\*

A number of review articles during recent years have been concerned with reports of interactions of phenols with surfactants or other macromolecules and the accompanying decrease in the efficiency of the phenols as preservatives (1, 2, 3). This article surveys some of the factors contributing to such interactions and considers the relation between the magnitude of interaction and preservative efficiency. It should be noted, however, that phenolic compounds are not the sole offenders among preservatives. In fact, almost all commonly used preservatives undergo similar interactions and subsequent inactivation to a greater or lesser extent. Fortunately, preservatives are generally employed considerably in excess of minimum effective concentrations and any interaction which occurs may not decrease the preservative availability to a level which permits microbial growth. Actual product breakdown may be encountered only when the interaction is particularly significant or when the interfering substance is present in relatively high concentration. The ultimate stability of a product in the field will of course depend on a great many variables such as quantity and nature of organisms introduced during the manufacturing and packaging, influence of agents in the formulation other than the primary preservative, and conditions under which the product

might be stored. Nevertheless, recognition of some of the factors contributing to preservative-macromolecule interactions might not only make possible the avoidance of some potential incompatibilities but should also aid in the selection of the most efficient preservative for a given system.

### Nature of Action

Phenols in general are denaturing agents and, if present in sufficient concentration, disrupt cell metabolism and are toxic to all types of cells. The work of Ferguson (4) demonstrated that for such structurally non-specific agents the antimicrobial activity would be proportional to the thermodynamic activity of the phenol in the medium. The thermodynamic activity determines the tendency for the drug to transfer from the solvent medium and enter the biological system and has also been referred to as the "availability" of the drug. Data of Ferguson and others indicate that as the phenol molecule is modified to cause it to have less affinity for the solvent medium the thermodynamic and antimicrobial activities can be expected to increase proportionately, at least over a limited range of modification (5). Thus, inhibitory concentrations of the p-hydroxybenzoate esters for the organism *Aspergillus niger*, under identical test conditions, have been reported as 0.1% for the methyl ester, 0.04% for the ethyl ester, and 0.02% for

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the propyl ester (6). Conversely, structural modifications which cause an increase in the affinity of the phenol molecule for the solvent may cause a corresponding decrease in the availability and effectiveness of the agent.

Likewise, any change in the solvent medium which results in an increase in affinity of solvent for the phenol will be expected to decrease the effectiveness of the phenol. Glycerin diminishes the germicidal effect of phenol in aqueous solution, and in fats the phenol is practically inert (7). This is an example of decrease in thermodynamic activity or escaping tendency for phenol in glycerin or fats due to increased affinity of solvent for the solute.

### Inactivation

The inhibition of phenolic preservatives has been the topic of several extensive review articles (1, 2, 3); therefore, only a few pertinent examples will be cited in this discussion.

It has long been recognized that the presence of protein interferes with the effectiveness of phenols. It has been demonstrated that phenol is adsorbed by protein (8), and thus the inhibition can be attributed to the tendency for the protein to compete with the bacterial cell for the available phenol.

Particular attention has been focused on the interaction of phenols with surface active agents, especially nonionic surface active agents. The activity which is typical of phenols in aqueous surfactant solutions has been demonstrated and discussed by Bean and Berry (9). In very dilute solutions of any surface active agent there is little or no surfactant-germicide interaction, although the activity of phenols in such solutions is often enhanced. Presumably this enhancement of activity is attributable to the interfacial activity of the detergent. At some higher concentration of detergent the activity of the antimicrobial agent is reduced as a result of the increasing tendency of the surfactant to bind the germicide and thus compete with the microorganism for the available germicide. This effect can be observed with many antimicrobial agents and surfactants of all types. The present discussion of surfactants will be limited to systems of relatively high concentrations of surface active agents such as might be encountered in cosmetic preparations. The inactivation is particularly evident in systems containing nonionic surfactants since the nonionics are often present in relatively large concentrations and they themselves have little or no inhibitory effect on the growth of microorganisms.

### Nature of Interaction Between Phenol and Macromolecule

When the mechanism of these interactions is discussed, reference is usually made to either micellar solubilization of the phenolic or to the formation of molecular complexes of the type described by Higuchi and co-workers (10, 11). It is unnecessary to attempt to distinguish between these mechanisms. Micellar solubilization and, indeed, micelle formation itself, can be considered to fall within the broad scope of the complex formation described by Higuchi.

Higuchi and Lach (10) indicated that compounds such as phenols, with acidic hydrogen, would be expected to associate with electrophilic atoms such as oxygen, and they indicated that such an association could be rendered favorable by the interaction or squeezing together of the hydrophobic portions of the molecules. The contribution of the squeezing together of the hydrophobic portions to the stability of the complex can be considered as arising from the entropy increase

which results from the liberation of water molecules associated with interacting molecules.<sup>1</sup> The over-all stability of the complex would then be a function of the energy of interactions such as the dipole-dipole interaction and of the entropy contribution resulting from the liberation of bound water. At room temperature the entropy contribution could be quite significant for a molecule with a large polar group (12).

A micelle of nonionic surfactant might be expected to provide an ideal model for association with a phenol—providing both the possibility for hydrogen bonding and also a non-polar portion. Molecules such as Carbowax,<sup>2</sup> having an ether function to participate in hydrogen bonding but without a large non-polar group, also show complex formation with phenols, but to a much lesser degree than do the surface active agents. On the other hand, marked similarity in the nature of binding of methylparaben by Tween 80<sup>3</sup>, a nonionic surfactant, and sodium lauryl sulfate, an anionic, suggest that the contribution of the hydrophobic bond is of considerable importance in this binding (13). Although the relative importance of the dipole-dipole interaction and the interaction of hydrophobic groups might vary considerably in the examples of binding discussed, there appears to be little necessity for attempting to attribute an interaction to one mechanism with exclusion of other possibilities.

Not only do hydrophobic groups in the macromolecule contribute to the degree of interaction, but increasing the hydrophobic character of a phenol molecule also results in increased affinity for the surfactant. Thus, the parabens have decreasing affinity for nonionics in the order: propyl > ethyl > methyl.

### Determination of Degree of Binding

Of greater significance than the exact mechanism for the interaction of the phenol and macromolecule is the availability of the phenol in the system. An equilibrium dialysis method can be a convenient means of determining the influence of a macromolecule on the availability of a preservative if there is available a dialysis membrane which is permeable to the preservative but not to the macromolecule. At equilibrium, the activity of the preservative will be identical on both sides of the membrane, and in dilute solution it can be assumed that the concentration of free preservative will be essentially equal on both sides of the membrane. If the macromolecule shows little or no direct influence on the growth of the microorganism, and if there are no other agents in the system which might influence the growth, it is to be expected that the effectiveness of the preservative might parallel the concentration of free preservative. Membranes of cellophane, nylon, and rubber have been employed for the studies discussed in this paper. Cellophane is satisfactory for larger molecules such as methylcellulose and Polyox<sup>4</sup>; nylon, which is of smaller pore size, can be employed to prevent the passage of Tween 80; and for molecules such as lower molecular weight poly(ethylene glycol) or small molecules such as propylene glycol it is necessary to use a membrane such as rubber.

(Continued on page 32)

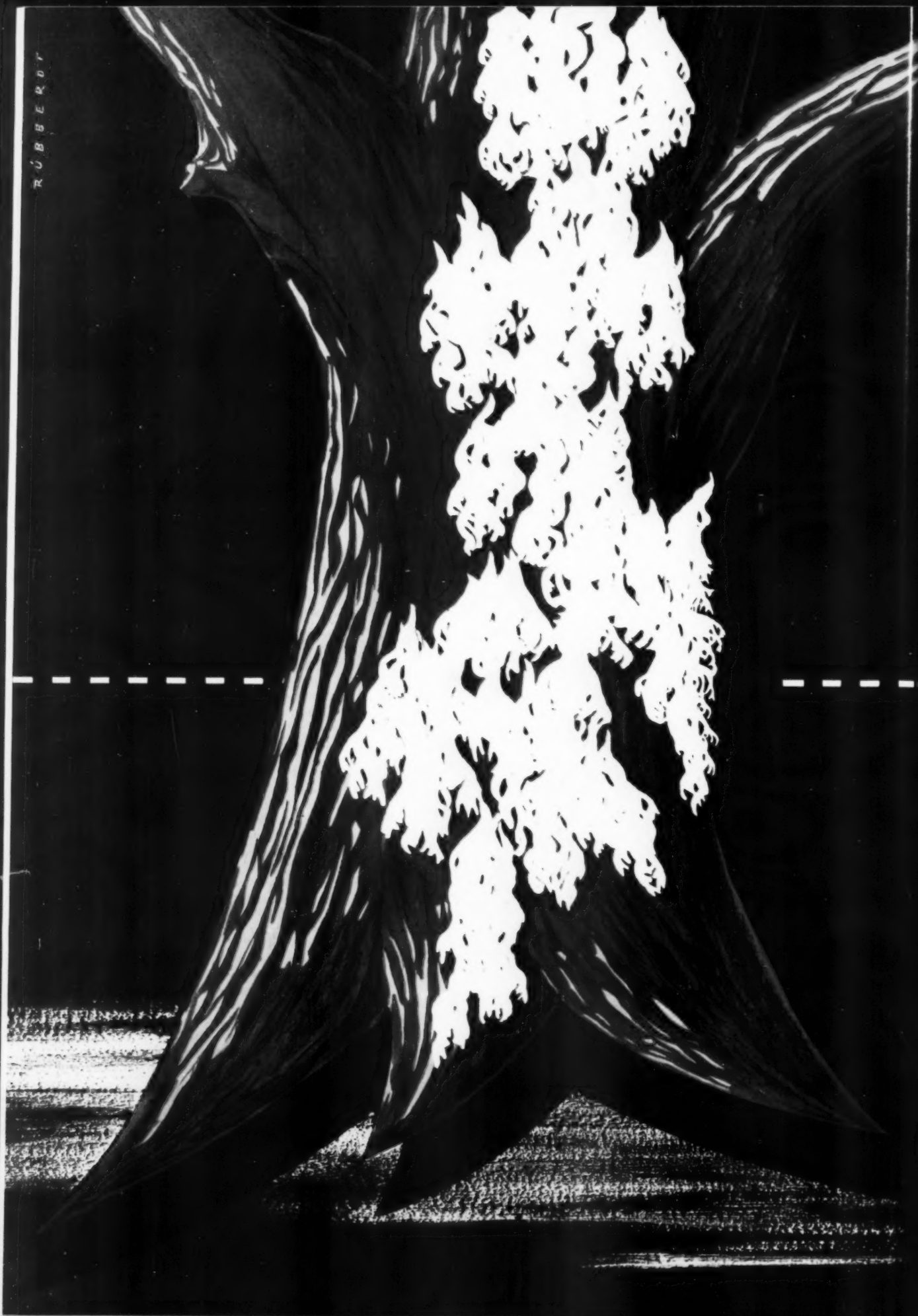
<sup>1</sup>Entropy can be considered as a measure of the disorder of a system. If before the interaction of two molecules one or both of the molecules has associated with it a number of water molecules, and on the interaction of the molecules the bound water is liberated, the result is a less ordered system and an increase in entropy. The free energy change for a reaction (determining the equilibrium position of the reaction) is related to entropy,  $S$ , and enthalpy,  $H$ , by the expression:  $\Delta F = \Delta H - T \Delta S$ . For a reaction to proceed spontaneously  $\Delta F$  must be negative.

<sup>2</sup>Carbowax® is poly(ethylene glycol), Union Carbide Chemicals Co., New York.

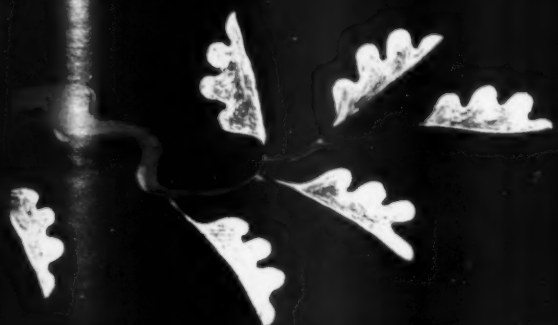
<sup>3</sup>Tween® 80 is polyoxyethylene 20 sorbitan monooleate, Atlas Powder Co., Wilmington, Del.

<sup>4</sup>Polyox® is a poly(ethylene oxide) of exceptionally high molecular weight, Union Carbide Chemicals Co., New York.









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An alternative technique would be a solubility method in which observations are made on solubility as a function of additive concentration. In such a study the preservative concentration is a constant, corresponding to the solubility in water, and any increase in solubility above this value must be due to association between the preservative and additive. A disadvantage of this approach is that the interaction is studied at but one preservative concentration. In some instances the interaction may be highly dependent on the concentration of the preservative, and a measure of the degree of interaction at saturation might not be a valid estimate of the magnitude of interaction at some lower concentration at which the preservative might normally be employed.

Other methods might be devised to study such interactions, the exact method depending on the properties of reactants and the magnitude of the interaction. Data obtained from these studies provide information on the concentration of free and bound preservative as a function of additive concentration.

#### Prediction of Required Preservative Concentration

Pisano and Kostenbauder (14) demonstrated that when varying concentrations of Tween 80 were added to an aqueous system containing methyl p-hydroxybenzoate the preservative activity of the methyl p-hydroxybenzoate was, as predicted, dependent on the concentration of free or unbound preservative. Under the conditions of these studies it was found that in the absence of Tween 80 a concentration of 0.075-0.080% methylparaben inhibited growth of *Aerobacter aerogenes*. When Tween 80 was added to this system in concentrations ranging from 2-6% it was found that the total quantity of paraben required to produce inhibition increased as the Tween concentration was increased, but in each case the concentration of free methylparaben required to inhibit growth was 0.065%. This concentration of free paraben is slightly less than the concentration of 0.075-0.080% which was noted for systems without Tween. The decrease in concentration of free paraben required might be attributed to the surface or interfacial activity of the surfactant and would be analogous to the enhancement in activity of preservatives when surfactants are present in such low concentrations that surface tension is reduced but there is little or no detergent-germicide interaction.

The above information provides a means to predict required preservative concentration from a knowledge of binding data. Since inhibition of microbial growth in absence of Tween can be obtained by a paraben solution of a thermodynamic activity corresponding to a concentration of less than saturation, it follows that there must be some paraben concentration less than saturation which will prevent microbial growth in the presence of the Tween. The equilibrium between free and bound preservative might be considered as follows:

$$\text{Preservative} + \text{Tween} = \text{Preservative-Tween}$$

By means of dialysis or solubility studies it is possible to determine the total and free preservative concentration in the presence of varying Tween concentration, and the ratio,  $R$ , of total/free preservative can be plotted as shown in Figures 1 and 2. If the required concentration of free paraben is known, the proper total amount of preservative to be added to the system can be obtained by selecting from the graph the  $R$  value corresponding to the concentration of surfactant present and then calculating as follows:

$$R \times \begin{matrix} \text{(Required Free)} \\ \text{(Preservative)} \\ \text{Concentration)} \end{matrix} = \begin{matrix} \text{(Required Total)} \\ \text{(Preservative)} \\ \text{Concentration)} \end{matrix}$$

It is not expected that the required free preservative concentration will generally be known with accuracy. It is suggested that as a first approximation it is permissible to use for required free preservative concentration the values which are normally recommended for solutions in which there are no interfering materials. These values are generally somewhat greater than the actual minimum inhibitory concentrations, and the calculated concentrations will probably be somewhat greater than the actual minimum value. The calculated value, therefore, sets an upper limit as a starting point for the formulator; and this information alone, which can be obtained within 24 hours, might save weeks of trial and error determinations. Of course, the final optimum concentration to be employed can best be determined through actual microbiological testing of the complete formulation. The  $R$  values indicative of the influence of representative macromolecules on methylparaben and phenol are presented in Figures 1 and 2, and range from those which might cause significant inactivation to those in which there is no measurable interference.

In addition to providing an approximate maximum concentration of preservative which might be required, these data are valuable in comparing the relative efficiency and economy of various agents. Figure 3 shows the relative affinity of methyl and propylparaben for Tween 80. While propylparaben might be effective in relatively low concentration in aqueous solution, the high degree of interaction of propylparaben with the

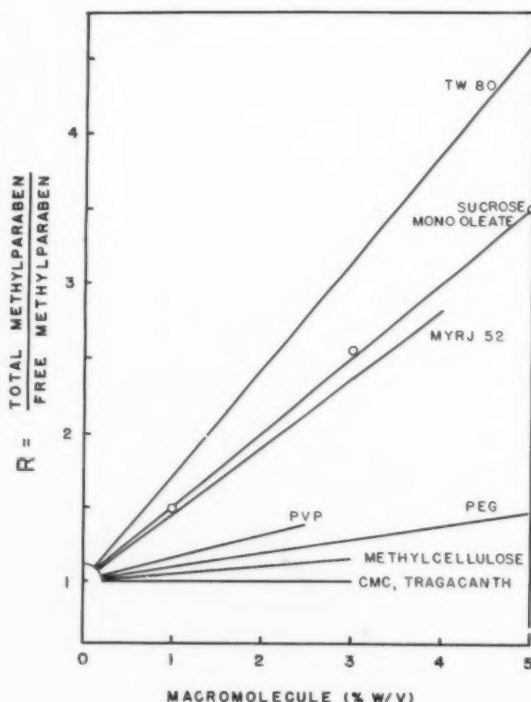


Fig. 1.— $R$ , the ratio of total/free methylparaben concentration as a function of concentration of various macromolecules.  $R$  value of 1.0 corresponds to no measurable binding. Data obtained from following references: (13,17,18,19).

<sup>1</sup>Myrj® 52 is polyoxyl 40 stearate, Atlas Powder Co., Wilmington, Del.

CMC® is cellulose gum (carboxymethylcellulose), Hercules Powder Co., Wilmington, Del.

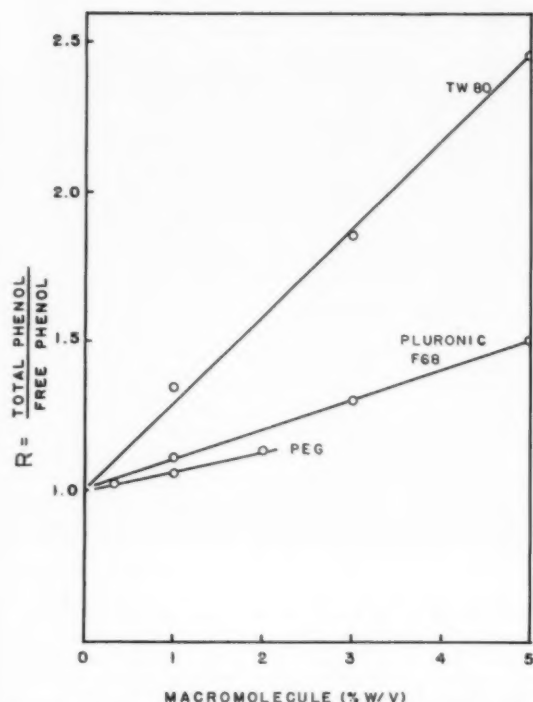


Fig. 2.— $R$ , the ratio of total/free phenol concentration as a function of concentration of several macromolecules.<sup>1</sup>  $R$  value has some dependency on phenol concentration; data presented here are for a total phenol concentration of 0.5%. Data from Lippman (13).

<sup>1</sup>Pluronic® F68 is a polyoxyethylene-polyoxypropylene surfactant, Wyandotte Chemicals Corp. Wyandotte, Michigan

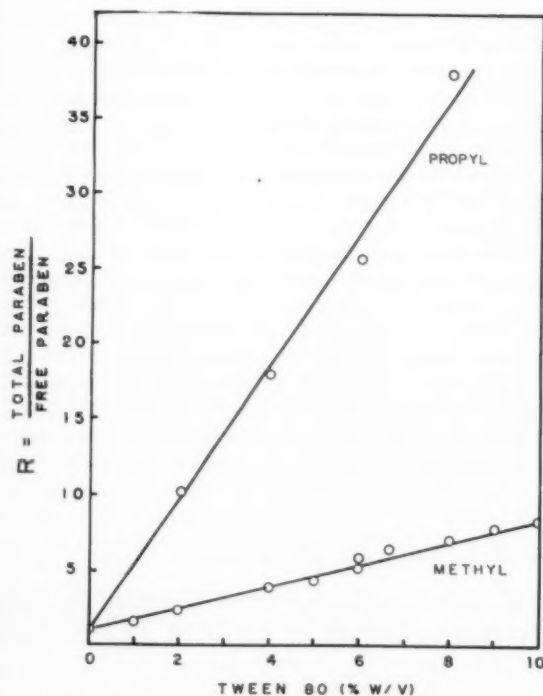


Fig. 3.—Relative affinity of Tween 80 for methyl and propyl esters of *p*-hydroxybenzoic acid. Data from Patel and Kostenbauder (17).

surfactant might make methylparaben the more efficient preservative in the surfactant solution.

In using the dialysis method no distinction need be made between mechanisms such as complex formation, micellar solubilization, or over-all solvent effect, since the availability of the preservative is measured directly. In complex systems, however, one must not lose sight of the fact that other components in the formulation might influence the required preservative concentration other than through association. Thus, while a combination of 0.18% methylparaben and 0.03% propylparaben failed to prevent bacterial and fungal growth in a mineral oil emulsion in which the emulsifier consisted of 7.5% Tween 80 and 2.5% Span 80, the same formulation containing 10% propylene glycol showed no microbial growth (15). Poprzan and deNavarre (16) investigated the influence of a number of glycols in systems of this type and found that when methylparaben and propylene glycol were used in combination in surfactant solutions it was possible to inhibit growth with concentrations which were not effective when the agents were used individually. Thus, while a 16% concentration of propylene glycol was required to inhibit *Aspergillus niger* in the presence of 2% of a nonionic, a combination of 8% propylene glycol and 0.2% methylparaben prevented growth of the organism. One of the possible explanations for these observations might be that the presence of various glycols enhanced the activity of the parabens by interfering with binding of the paraben to the surfactant. Dialysis studies with a rubber membrane, which is impermeable to both Tween and the glycol, show that the presence of the glycol does not increase the availability of the paraben in the system and therefore the effectiveness must be attributed to some factor other than modification of the surfactant-germicide interaction (13).

A further consideration in these preservative-macromolecule interactions is the possibility of the formation of a relatively insoluble complex. Such a situation would not only inactivate the preservative but would also influence the emulsifying or suspending properties of the macromolecule. In all of the systems considered in this discussion, it would appear that the resulting association product is sufficiently soluble to permit the attainment of effective preservative concentrations. It is likely that in the great majority of examples required preservative concentrations can be obtained without precipitation, even though precipitation might be known to result at some higher concentration. ■

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1959 AEROSOL PACKAGING AWARDS WINNERS



GEORGE W. FIERO

C. S. M. A.

## 46TH ANNUAL MEETING



"OH LA LA Eau de Parfum Mist Concentre," a product of Parfums Ciro, Inc., New York, N. Y. received the top award as the most attractively designed package entered in the Perfumes, Colognes and Toilet Waters category of the 1959 Aerosol Packaging Awards Competition.

George W. Fiero of Esso Standard Oil Company was elected President of the Chemical Specialties Manufacturers Association, at this organization's 46th Annual Meeting in Washington, D.C.

H. W. Hamilton, for many years Secretary of the Association, was appointed to the newly created post of Executive Vice President.

Other Officers elected were First Vice-President, Charles E. Beach, John C. Stalford & Sons Inc., Baltimore; Second Vice-President, Charles E. Alderdice, Jr., The Bell Company, Chicago; Treasurer, Frederick G. Lodes, Lodes Aerosol Consultants Inc., New York; and Secretary, A. A. Mulliken.

Elected to three-year terms on the Board of Governors were Donald M. King, Masuryoung Co., Boston; Peter C. Reilly, Reilly Tar & Chemical Corporation, Indianapolis; Joseph E. Lee, McLaughlin Gormley King Co., Inc., Minneapolis, and Joseph J. Tomlinson, General Chemical Division, Allied Chemical Corporation, New York.

Abstracts of some of the papers follow:

### CHEMICAL SENSIBILITY TO THE ENVIRONMENTAL ASPECTS OF AIR CONDITIONED AREAS

John von Bergen, Airkem, Inc.,  
New York, N. Y.

Chemical sensibility is primitive; the most lowly of animals possesses it—long before they develop





A PANEL OF SIX JUDGES examined the 269 entries in this year's Aerosol Packaging Awards Competition to select the top design in each of eleven product categories plus one of the eleven as the GRAND AWARD winner. Seated are Mrs. Fred C. Poppe, New York Housewife, who holds the Grand Award winner, "QWIP," a dessert topping which also won top award in the Food Products division, and Mrs. Charlotte Montgomery, Associate Editor of Good Housekeeping Magazine. Standing, left to right, are Charles R. Beall, Vice President-Trade Promotion, McKesson & Robbins, Inc., William Pahlmann, interior designer and President of William Pahlmann Associates, Inc.; and Walter F. Kohn, marketing consultant. Absent from the photograph is Lawrence Drake, Editor, Grocery Edition, Chain Store Age magazine.

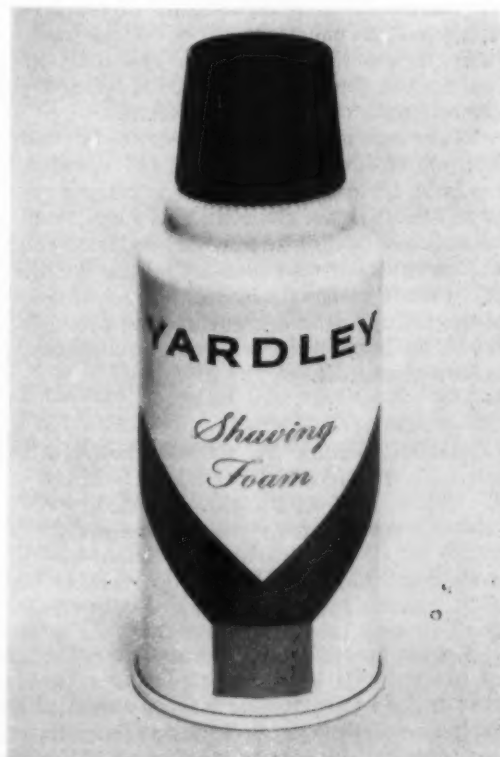


"SPRAY AND WAVE," hair spray, a product of Cort Cosmetics, Inc., Stamford, Conn., received the top award as the most attractively designed package entered in the Hair Preparations category of the 1959 Aerosol Packaging Awards Competition.

brain, nervous system, eyes, ears, nose or mouth. It may be taste or it may be smell but in such primitive creatures, we can only be sure that their behavior is changed by chemicals introduced into their environment. The eventual improvement through evolution modifies the perceptual apparatus of animal life. There is a gradual but immense development of chemical senses usually characterized by the following features: (1) Reduction in size of the sensitive areas. (2) Sensations become progressively more sharply defined.

In man, the surfaces sensitive to smell are similar in character and as sensitive as the skin of the primitive sea creatures. Although the act of perceiving odors is a relatively simple process, the perceptual pattern induced by the perceived odor on the other hand is indeed complex. The sensory and developed impulses resulting in the final pattern are usually described as physical, chemical, physiological. The two primary needs of man—nutrition and reproduction—were both at one time aided by the apparatus of odor sensation transmission. Our modern way of life is altered and this major sensing apparatus has been neglected. However, the sense of smell still lives strong within man and is a most important function in our environment.

Other aspects of environmental control in air conditioned areas are the problems of airborne bacteria and the unbalance of positive and negative ions in the atmosphere. Recent methods of control are discussed with strong emphasis on the discipline for control.



"YARDLEY SHAVING FOAM," a product of Yardley of London, Inc., Union City, N. J., received the top award as the most attractively designed package entered in the Shave products category of the 1959 Aerosol Packaging Awards Competition.

In addition to a brief review of temperature and humidity factors as they might affect environmental comfort, the author presents the inter-relations between temperature, humidity, threshold odors, subliminal perception of odors, the relation of airborne bacteria count to incidence of reinfection, and the combined effect of these factors which may produce unusual health patterns in occupied air conditioned areas.

Physiological and psychological problems are discussed as well as available solutions to these problems. A summary of challenges and recommendations for the future will be presented.

#### **MODERN CLEANING METHODS IN THE HOME**

**David Justice, Lever Brothers Company,  
Edgewater, New Jersey**

Throughout history mankind has paid special attention to cleanliness in the kitchen, even before the development of bacteriology and the growth in our knowledge of microbiological phenomena.

In the past century, scientific investigations of food spoilage and pathogenic organisms have supplied the information needed for establishing valid standards of cleanliness. The careful washing of dishes and the thorough scrubbing of counters, table tops and floors are not only desirable from an aesthetic viewpoint, but they also remove the substrate needed by micro-organisms for growth. The chemical processing industry has developed new products designed to achieve real cleanliness, utilizing the results of many decades of research. These new products are more effective and easier to use than the old fashioned ones. The most important tool used in the development of these new products are scientific evaluation procedures.

These testing methods, in order to be really useful in product development, must be reproducible and realistic. Unfortunately, there is often a conflict between these goals. Reproducible results can usually be obtained under somewhat artificial conditions; the results of tests under actual use conditions are often made uncertain by random uncontrolled variables. Future development of new products will be based on improved evaluation procedures, as well as new chemicals.

#### **COMMON DISEASES COMMUNICABLE FROM HOUSEHOLD PETS TO MAN**

**R. L. Burkhardt, V.M.D.,  
American Cyanamid Company,  
Agricultural Division,  
Animal Health Department,  
Princeton, New Jersey**

The purpose of this paper is to describe infections of pet animals in the United States that may be transmitted to man. The most common of these infections will be covered first, and those that are less common will be briefly mentioned. The infections will include viral, bacterial, fungal, and helminth. Since the problem of controlling these infections varies mainly according to the type of organism,

rather than the host species, the subject will be dealt with accordingly.

Of the animals commonly regarded as pets, most is known about the infections of the dog, cat and parakeet. However the problem of disease transmission extends to laboratory animals as well, and in this class of animals we will include the mouse, rat, hamster, rabbit and monkey. Where possible, the mode of transmission of the infecting agent will be covered with comments on the degree of difficulty in killing them with chemical agents.

#### **Status and Future of Aluminum Aerosol Containers**

**John M. Jackson,  
American Can Company,  
Research Division,  
Barrington, Illinois**

Some background is given concerning previous consideration of aluminum as a can making material in the United States and Europe. The advantages of the extruded aluminum container in regard to appearance, strength, etc., are listed, and some of the problems associated with the use of aluminum are also listed.

Fabrication of the container is described. The characteristics of the container are discussed in regard to: strength, corrosion resistance and the role of internal organic coatings.

#### **THE EFFECTS OF FLUOROCARBON PROPELLANTS AND METHYLENE CHLORIDE ON VARIOUS PLASTICS**

**James A. Brown, General Chemical Division,  
Allied Chemical Corporation,  
Technical Service Laboratory,  
Edgewater, New Jersey**

The Effects of Fluorocarbon Propellants & Methylene Chloride On Various Plastics. The physical changes that occur to various plastics after contact with fluorinated hydrocarbons for comparatively prolonged periods of time have been determined. Of prime interest were the dimensional and weight changes of the plastic specimens. Tests were also made using methylene chloride so that comparisons could be made with a representative chlorinated hydrocarbon. These tests were made essentially in accordance with ASTM test D-543-52T.

In general it was found that in substituted methanes swelling is more pronounced with solvents having hydrogen in the molecule, such as methylene chloride and Genetron 21, than completely halogenated molecules such as Genetron 11 and 12. In substituted ethanes the swelling becomes less as chlorine atoms are replaced by fluorine.

#### **THE POTENTIAL FOR THE AEROSOL MARKET**

**Ralph A. Crane, Market Research Manager,  
"Freon" Products Division,  
E. I. duPont de Nemours & Company, Inc.,  
Wilmington, Delaware**

In the next ten years, four times as many nonfood aerosols—about eight billion—will be produced and

sold as in the last ten years. And although food products new represent less than 20% of the total pressure-packaging market, they may become the single largest growth area in the next decade. This optimistic forecast is based on the predicted growth of the United States market, the growth rate to date of the aerosol industry, an awareness of the reasons why the aerosol package has been so successful, and market saturation figures for various aerosol products.

Six principal products—insecticides, room deodorants, shave creams, hair sprays, surface coatings, and colognes—accounted for 81% of the 1958 total production of 485 million non food packages. Additionally, about 80 million food aerosols, principally whipped toppings, were produced last year. Sales of today's principal nonfood products will continue to expand, and together with the sales of dental cream they should again dominate the 1968 nonfood aerosol market of one billion units.

Despite the advances gained to date by pressurized food products, there are still many formidable problems of food preservation, purity and sanitation. The success of pressure-packed whipped cream however, indicates that the problems are not insurmountable. Newly developed "Freon-C318" propellant, which is odorless, tasteless and chemically stable, should help the aerosol food market grow. The challenge of the next decade is two billion nonfood and food aerosols per year. Whether or not this goal is attained depends on the further successful development of food aerosols.

#### PRODUCTION OF CHEMICAL SPECIALTIES

Herbert L. Green and Julius A. Neuberger,  
Burlin & Company, Inc.,  
Indianapolis, Indiana

The paper discusses the production of chemical specialties for other than retail markets. Included are various aspects concerning plant location, manufacturing equipment, production schedules, storage and handling of materials, labor, housekeeping, and quality control.

#### RECENT DEVELOPMENTS IN INSECT ATTRACTANTS

Morton Beroza  
Entomology Research Division,  
Agricultural Research Service,  
U. S. Department of Agriculture,  
Beltsville, Md.

Certain odors evoke so compelling a response in insects that they appear to have little choice but to seek out the source. Entomologists and chemists of this Division have teamed together to find chemicals that emit such attracting vapors because these materials can be used to combat insects of economic importance.

In actual practice lures, specific for the males of a certain species, are used in traps to detect incipient infestations of that species and later to check the progress of control measures that are applied. With the aid of this technique the Mediterranean

fruit fly (Medfly) was eradicated rapidly from the State of Florida. Scientists of this Division are also investigating the use of lure-toxicant combinations for direct control of insects.

Our most fruitful approach to the finding of insect attractants has been via volume screening of chemicals followed by synthesis of compounds related to the best lures found. By this means we discovered *siglure* (sec-butyl 6-methyl-3-cyclohexene-1-carboxylate), the attractant used in the Florida Medfly campaign, and recently two better attractants, the *sec*- and *tert*-butyl esters of 4 (or 5)-chloro-2-methylcyclohexanecarboxylic acid. The *sec*-butyl ester, which we named *medlure*, is about twice as attractive and about five times as persistent as *siglure*. The *tert*-butyl ester, while even more attractive to the Medfly than *medlure*, is unfortunately difficult to make in good yield.

Newer finds are 4-(*p*-acetoxypheyl)-2-butanone and several related chemicals, which attract the melon fly, and 1,2-hexadecanediol and 1,2-epoxyhexadecane, gypsy moth attractants.

Studies of chemical structure as related to attractancy of lures and commercial possibilities of chemical lures will be discussed.

#### MOLD POPULATIONS WITHIN THE HOME

Mary A. Swaebly-Ehrlich, Ph. D.,  
Duquesne University, Department of Biology,  
Pittsburgh, Pennsylvania

Fungi are considered by many allergists to be second in importance only to pollen as excitants of respiratory allergy. House dust and furniture stuffing have long been known to contain allergenic substances and both are also known to harbor a variety of molds. As a consequence, many theories regarding the possible relationships between household fungi and respiratory allergy have been proposed.

In the present investigation dust samples from nearly 100 homes were analyzed for the numbers and kinds of culturable molds present. The air within 15 feet of these same homes was sampled for fungi content at various times throughout one day, such sampling being repeated at intervals over a two month period in several of the homes. In addition, several types of new and used furniture stuffing were assayed for quantitative and qualitative mold determinations.

The results of this survey clearly show that each home must be regarded as a separate entity. The mold population of the outside air follows a fairly predictable seasonal trend characteristic for a given area, but there is no apparent relationship between the numbers and kinds of organisms present in the air outside of the home with those present inside the home. The degree of activity within the home at any given time largely determines the number though not the kinds of organisms present in the air. Both new and used furniture stuffing materials, including foam rubber, contain many fungi. Of additional and special interest is the fact that as furniture is subjected to use, the kinds as well as the number of organisms in the stuffing may change.



## APPLICATION OF PENDANT DROP MEASUREMENTS TO EMULSION POLISHES

George J. Fuld, Department of Food Technology,  
Massachusetts Institute of Technology,  
Cambridge, Massachusetts

and  
Melvin Fuld,  
Fuld Brothers, Inc.,  
Baltimore, Maryland

The prediction of the ability for a "dry bright" floor polish to spread on a flooring surface is a complex problem. Using the Harkins' equation, it may be shown that spreading is a function of the surface energy of the flooring surface, the interfacial surface tension between the liquid and the surface tension of the fluid. This predicts that spontaneous spreading will occur if the surface energy of the solid is greater than the sum of the surface tension of the spreading fluid and the interfacial tension of the fluid and the surface. The difficulties in measurement of these quantities are compounded by the fact that no accurate measure of the surface energy (interfacial energy of the solid surface and air) has been developed as well as the variable effect of porosity and smoothness of the surface. In addition, the liquid surface tension and liquid interfacial tension to the solid are dynamic properties which vary with time after formation in the complex emulsion system of a floor polish.

The application of the "pendant drop" method to the measurement of surface tension can measure the dynamic changes of a floor polish surface tension. Typical measurements of different polish systems are shown. The possible effect of the surface tension changes with time on the polish spreading ability are discussed.

## MARKETING OF CHEMICAL SPECIALTY PRODUCTS

Dr. Edwin I. Stoltz, Technical Director,  
Adell Chemical Company, Holyoke, Mass.

Over 28 billion chemical specialty packages are sold annually. This includes both the retail consumer and the wholesale industrial markets. Dr. Stoltz's remarks are slanted to the household consumer field.

The three essential functions of marketing are Advertising, Selling and Distribution. The paper deals with the company's policies on advertising and selling.

### ADVERTISING

Major amount of advertising budget is allocated to television. All commercials are made within the following framework based on company advertising policies:

- 1) All commercials or other advertisements must be factual.

Films never make exaggerated claims which products cannot live up to. The firm has found that in many cases the customer, after finding that a product works for a particular job, will experiment and come up with his own uses for the product.

- 2) No comparisons should be made with another product or products.

Company is firmly convinced that the public deplores excessive comparison of competitive products.

- 3) All statements must be of a positive nature. Claims are accurate and are presented with hard sell. Time is not wasted with non-essentials.
- 4) The approach is soft so that it will not antagonize the listener.

Demonstration type of commercial has been found least antagonizing. Also this approach has been found far better than merely describing the product.

### SELLING

The company has been selling chemical specialty products for twenty-six years. Since the early days of Lestoil, the company has maintained the belief that each participant in the selling of its products should receive a good profit. This is the heart of selling chemical specialties. The framework governing sales policies is:

- 1) Salesmen are cautioned against making comparisons with other products.
- 2) There is no high pressure selling to load a quantity of merchandise on a customer.
- 3) Fair trade is maintained, and there is no price cutting or deviation from the established price.
- 4) No free goods.

The buyer always asks for a deal, in the form of free goods. The company's policy is that there are no free goods or deals at any time. Money which might be used for this purpose is allocated to advertising.

- 5) There is one price for all.

This point can be badly abused in selling chemical specialties especially on the industrial level. This type of selling is a discredit to the industry.

## THE EFFICIENCY OF IODINE ON SURFACES

Bernard Witlin and Louis Gershenfeld,  
Department of Bacteriology,  
Philadelphia College of Pharmacy and Science,  
Philadelphia, Pennsylvania

Iodine sanitizing solutions were prepared separately, using Iodine Solution (2% free iodine) and various Iodophors to give solutions with varying free-iodine concentrations.

The surfaces of sterilized one inch squares of steel, ceramic tile, wood and linoleum were inoculated in each instance with a mixture of suitable test organisms. Each of the inoculated squares was exposed to a solution of a known free iodine concentration. After the desired exposure, the treated inoculated squares were cultured, the medium containing an iodine-inactivator. The findings of these cultures were recorded.

In all instances, solutions containing 25 p.p.m. of free iodine rendered all surfaces free of the test organisms within 5 minutes.

(Continued on page 54)



# The Synthesis and Properties of Mercaptans Having Different Degrees of Acidity of the Sulfhydryl Group

J. W. HAEFELE and R. W. BROGE\*

IN A RECENT PAPER, Voss (1) discussed the skin sensitizing properties of a number of water-soluble mercaptans. Voss' study mentioned many compounds new to the literature. The major purpose of the present paper is to describe the synthesis and properties of these new mercaptans. A subsequent paper will give the behavior of these mercaptans toward hair.

Our intent in this mercaptan work was to design and prepare compounds in which the acidity of the sulfhydryl group was widely varied. This work arose in the first instance from our observation in 1952 of greatly increased acidity of the mercaptan group when thioglycolic acid was merely converted to thioglycolamide. There followed an extensive examination of all types of related compounds, including: esters, hydrazides, mono- and disubstituted amides, and some special cases such as mercaptoethyl acetamide. Later, other N-substituted mercaptoethylamine derivatives were studied, including compounds with SH in the quaternary cation, and compounds with SH acidity promoted by the introduction of  $CF_3$ . A four-thousand fold variation in acidity was achieved. This will be discussed below.

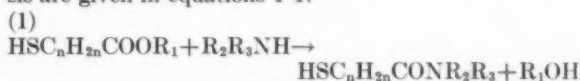
The major synthetic work was directed toward exploring two types of water-soluble compounds:

- Thioglycolic acid derivatives,  $HSCH_2COY$  and  $HSCHRCOY$ , where Y completes an amide, hydrazide, imide, or ester.
- Mercaptoethylamine derivatives,  $HSC_2H_4NR_1R_2$ .

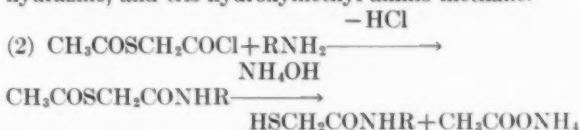
The preparative reactions will be discussed in general terms. Detailed representative examples are given in the experimental section.

## Mercapto Alkanoic Acid Derivatives

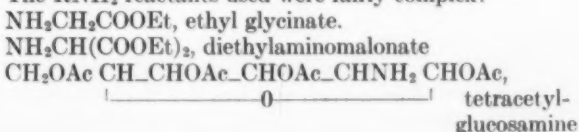
The parent compound was studied, and analogs were prepared (Table I). The most useful methods for synthesis are given in equations 1-4:



This represents the reaction of an ester with primary and secondary amines. Reactions in most cases proceeded to high yields. The volatile alcohol was driven off, and the residue purified, by distillation if possible. In the experimental portion, examples of some modified techniques will be given. The most useful esters were ethyl thioglycolate and methoxyethyl thioglycolate. There was a wide variation in the amines used, including methylamine, octylamine, monoethanolamine, di-ethanolamine, hydrazine, and tris hydroxymethyl amino methane.



The  $RNH_2$  reactants used were fairly complex:



\*The Procter & Gamble Company.

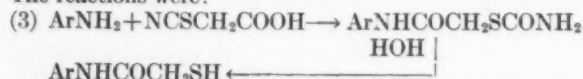
Reprinted from the Proceedings of the Scientific Section of The Toilet Goods Assn., Number 32, December, 1959.

**TABLE I**  
**Mercapto-Alkanoic Acid Derivatives of the Formula HSCHRCOY, where Y completes an Amide, Hydrazide, Imide or Ester**

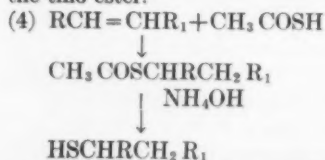
Formula	Method of Equation	(%) Yield	% Purity by Thiol Titration	B.p. or M.p. °C	% Sulfur Calc'd	% Sulfur Found	% Nitrogen Calc'd	% Nitrogen Found	pK
<b>Amides</b>									
N-Methylthioglycolamide	HSCH <sub>2</sub> CONHCH <sub>3</sub>	1	78.3	99.8	91.4° (5mm.)	30.50	30.60	13.30	12.90 8.05
Disulfide					124°	30.82	31.24	13.46	13.19
N,N-Dimethylthioglycolamide	HSCH <sub>2</sub> CON(CH <sub>3</sub> ) <sub>2</sub>	1	73.3	96.6	75-8° (5mm.)	26.90	26.40	11.80	11.10 8.10
N-Ethylthioglycolamide	HSCH <sub>2</sub> CONHC <sub>2</sub> H <sub>5</sub>	1	63.5	100.0	92-3° (4mm.)	26.90	27.00	11.80	11.02 8.14
Disulfide					101°	27.10	27.56		
N-Isopropylthioglycolamide	HSCH <sub>2</sub> CONHC <sub>3</sub> H <sub>7</sub>	1	6.0	92.2	112° (4mm.)	24.20	23.68	10.60	10.58
Disulfide					94°	19.90	20.48	8.69	8.26
N-Amylthioglycolamide	HSCH <sub>2</sub> CONHC <sub>5</sub> H <sub>11</sub>	1	6.0	97.9	131° (4mm.)	20.00	20.14	8.75	8.20
Disulfide					113°	18.32	18.32	7.99	7.56
N-Hexylthioglycolamide	HSCH <sub>2</sub> CONHC <sub>6</sub> H <sub>13</sub>	1	70.5	96.6	134° (2mm.)	18.42	18.48		
Disulfide					109°				
N-Decylthioglycolamide	HSCH <sub>2</sub> CONHC <sub>10</sub> H <sub>21</sub>	1	72.0	91.0	163° (0.5mm.)	13.94	14.43	6.09	5.70
Disulfide					118°	23.75	23.61	10.45	10.25 8.10
N-Hydroxyethylthioglycolamide	HSCH <sub>2</sub> CONHC <sub>2</sub> H <sub>4</sub> OH	1	96.1	81.5	86°	21.50	21.74	9.39	8.80 8.10
Disulfide					115° (2mm.)	16.80	16.10	14.70	14.66
N-Methoxyethylthioglycolamide	HSCH <sub>2</sub> CONHC <sub>2</sub> H <sub>4</sub> OCH <sub>3</sub>	1	51.3	96.3					
N-(Diethylaminoethyl) thioglycolamide	HSCH <sub>2</sub> CONHC <sub>2</sub> H <sub>4</sub> N(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	1	36.6	94.1	122-5° (2mm.)				
N,N-bis (2-Hydroxyethyl) mercaptoacetamide	HSCH <sub>2</sub> CON(C <sub>2</sub> H <sub>4</sub> OH) <sub>2</sub>	1		86.0					
N-(1,1-dimethylolmethyl) mercaptoacetamide	HSCH <sub>2</sub> CONHC(CH <sub>3</sub> ) <sub>2</sub> (CH <sub>2</sub> OH) <sub>2</sub>	1		86.0					
N-trimethylolmethyl mercaptoacetamide	HSCH <sub>2</sub> CONHC(CH <sub>3</sub> ) <sub>3</sub> (CH <sub>2</sub> OH) <sub>3</sub>	1		66.0					
N-carboxymethyl thioglycolamide	HSCH <sub>2</sub> CONHC <sub>2</sub> COOH	2		100.0					
N-dicarboxymethyl thioglycolamide	HSCH <sub>2</sub> CONHC(COOH) <sub>2</sub>	2		103.0					
N-mercaptoacetyl glucosamine	HSCH <sub>2</sub> CONHC <sub>6</sub> H <sub>11</sub> O <sub>5</sub> <sup>a</sup>	2		69.0					
2-Mercaptopropionamide	CH <sub>3</sub> CHSHCONH <sub>2</sub>	1		93.0					
3-Mercaptopropionamide	HSCH <sub>2</sub> CH <sub>2</sub> CONH <sub>2</sub>	1		91.0					
3-Mercaptopropionethanolamide	HSC <sub>2</sub> H <sub>4</sub> CONHC <sub>2</sub> H <sub>4</sub> OH	4		88.0					
<b>Hydrazides</b>									
Thioglycolhydrazide	HSCH <sub>2</sub> CONHNH <sub>2</sub>	1	96.5	94.2	52-3°	30.24	27.96	26.42	28.07 8.05
Disulfide					93-4°	30.52	31.32	26.68	25.32
2-Mercaptopropionhydrazide	CH <sub>3</sub> CHSHCONHNH <sub>2</sub>	1		88.0					
3-Mercaptopropionhydrazide	HSCH <sub>2</sub> CH <sub>2</sub> CONHNH <sub>2</sub>	1		96.0					
<b>Imide</b>									
Thiomalimide	—NH— COCH <sub>2</sub> CHSHCO	4		89.0					
<b>Esters</b>									
Hydroxyethyl Thioglycolate	HSCH <sub>2</sub> COOC <sub>2</sub> H <sub>4</sub> OH			99.4	86-91° (20-30mm.)				
Dimethoxyethyl Thioglycolate	HSCH <sub>2</sub> COOC <sub>2</sub> H <sub>4</sub> OCH <sub>3</sub>			99.2	130-134° (0.5mm.)				
	CH <sub>3</sub> COOC <sub>2</sub> H <sub>4</sub> OCH <sub>3</sub>								
	NHCOCH <sub>2</sub> SH								
	<sup>a</sup> CH <sub>2</sub> OH CHCHOH CHOH C CHOH								

It will be evident that in each case, other groups were protected so that reaction would proceed only between amine and acid chloride. Reaction was conducted in cold ether. Ethereal solutions of the two reactants were added to the reaction vessel at the same rate from separate funnels, to minimize reaction with the second amine hydrogen. The reaction proceeded readily. The product was crystallized and characterized, then dissolved in ammonia and water to form the mercaptan, which was not itself isolated. Instead, the solution was titrated for thiol content and diluted to concentrations desired for the studies on hair.

Certain aromatic mercapto-amides prepared by Weiss (2) were duplicated by us, but no new ones were made. The reactions were:



Another reaction used was the addition of thioacetic acid to an olefinic double bond followed by hydrolysis of the thio ester.



The addition reaction usually proceeded readily, but it was promoted with heat, and traces of base or benzoyl peroxide, where necessary. The mercaptans were released

in ammonia from the S-acetyl derivatives.

Two water-soluble esters (Table I) were prepared by removal of water from the alcohol-acid mix using a Dean-Stark tube and p-toluene sulfonic acid catalyst. The esters were distilled under reduced pressure.

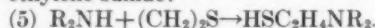
The compounds prepared are given in Table I.

The reaction to prepare the trimethylolmethyl compound was slow. At 66% conversion, the reaction mixture was diluted to the appropriate concentration and pH for sensitization studies.

Some difficulty was experienced in the hydrolysis of the mercaptoglucosamine, although the precursor was pure.

### Mercaptoethylamine Derivatives

The parent compound is mercaptoethylamine, HSC<sub>2</sub>H<sub>4</sub>NH<sub>2</sub>. This is a solid because of inner salt (3) formation. Many of the analogs prepared were liquids (Table II). The most important method employed (4) ethylene sulfide:



The reaction (5) is clean-cut to give excellent yields with secondary amines. The reactants are combined and heated, and the reaction mixture is distilled. Amines used included morpholine, pyrrolidine, piperidine, and 1-amino-2-diethylamino ethane, NH<sub>2</sub>C<sub>2</sub>H<sub>4</sub>NEt<sub>2</sub>. The morpholinium and pyrrolidinium salts were prepared from the above mercaptans by addition of CH<sub>3</sub>I. These are the first heterocyclic quaternary mercaptans that have been reported. These two compounds were surprisingly toxic. Intradermal injection of 0.10 ml. of a 0.19 M solution of



Seven years of original research have been rewarded by the discovery and development of a new group of controlled and reproducible perfume compositions for cosmetic and pharmaceutical products. We call them CHEMODERMS, for their chemical purity and dermatological safety. Exhaustive tests prove them free of primary irritants. Leaders in the treatment of allergies hail them as an important step in the elimination of sensitivity to perfumes. Now available in ten of the most desirable, panel-accepted fragrances. CHEMODERMS will enhance your next cosmetic creation, enlarge your markets, and enliven your presentations and promotions with vital and timely appeals.

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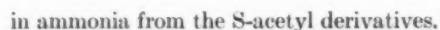
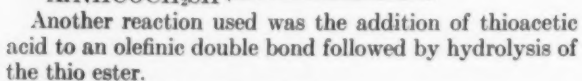
**FIRMENICH & CIE**  
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GENÈVE SUISSE

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FIRMENICH OF CANADA, LIMITED,  
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TABLE I

[illegible]

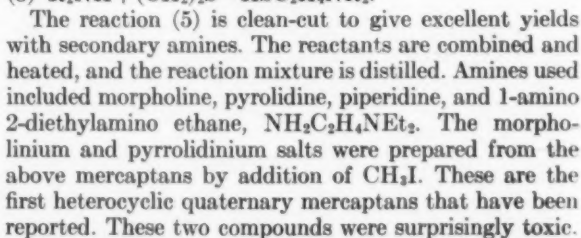
Certain aromatic mercapto-amides prepared by Weiss (2) were duplicated by us, but no new ones were made. The reactions were:



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For Finer Fragrance . . .  
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TABLE II  
Mercaptoethylamine Derivatives

Formula	Method of Equi- tion Yield	% Purity by Thiol Titra- tion	B.p. or M.p., °C.	% Sulfur Calc'd Found	% Nitrogen Calc'd Found	pK	n <sub>D</sub> <sup>20</sup>	M Found
<b>MERCAPTO AMINES</b>								
N-Mercaptoethylpyrrolidine	HSC <sub>2</sub> H <sub>4</sub> NC <sub>4</sub> H <sub>8</sub>	5 74.2	96.5	81 (23mm.)	24.48 24.76	10.68 10.35	1.5004	39.51 39.28
N-Mercaptoethylpiperidine	HSC <sub>2</sub> H <sub>4</sub> NC <sub>5</sub> H <sub>10</sub>	5 75.8	96.9	88 (18mm.)	22.09 22.32	9.66 9.03	1.5011 a	44.13 44.06
N-Mercaptoethylmorpholine	HSC <sub>2</sub> H <sub>4</sub> NC <sub>3</sub> H <sub>7</sub> O	5 77.9	98.8	72-3 (0.9mm.)	21.80 22.19	9.52 9.18	1.5045 b	41.05 41.01
N-Mercaptoethylpyrrolidone	HSC <sub>2</sub> H <sub>4</sub> NC <sub>4</sub> H <sub>7</sub> O	4	104.0*					
3-Aza-6-oxaheptanethiol	HSC <sub>2</sub> H <sub>4</sub> NHC <sub>3</sub> H <sub>6</sub> OCH <sub>2</sub>	5 46.6	97.8	57 (3mm.)	23.75 24.16	10.36 9.13	1.4770	38.38 38.20
3-Aza-5-diethylaminopentanethiol	HSC <sub>2</sub> H <sub>4</sub> NHC <sub>2</sub> H <sub>5</sub> N(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	5 17.0	98.4	53 (0.3mm.)	18.19 17.92	15.90 15.46	1.4793 c	
<b>MERCAPTO AMIDES, UREAS</b>								
N-Mercaptoethylacetamide	HSC <sub>2</sub> H <sub>4</sub> NHCOCH <sub>3</sub>	6 83.2	99.6	102 (0.7mm.)	11.86 11.83	27.17 26.93	8.22	1.5120 d
Disulfide				89-90	27.15 27.40	11.85 11.74		
N-Mercaptoethyltrifluoroacetamide	HSC <sub>2</sub> H <sub>4</sub> NHCOCF <sub>3</sub>	7 9.1	98.5	57-61 (1.2mm.)	18.53 19.38	8.09 8.46	7.30	1.4319 e
N-Mercaptoethylurea	HSC <sub>2</sub> H <sub>4</sub> NHCONH <sub>2</sub>	8	97.0					
<b>QUATERNARY DERIVATIVES</b>								
N-Mercaptoethyl-N-methylpyrrolidinium iodide f	HSC <sub>2</sub> H <sub>4</sub> N(C <sub>4</sub> H <sub>8</sub> )(CH <sub>3</sub> ) I	5 24.4	99.7	66-7*	11.75 11.81	5.13 5.15	7.70	
Disulfide				238	11.78 11.70	5.15 5.03		
N-Mercaptoethyl-N-methylmorpholinium iodide	HSC <sub>2</sub> H <sub>4</sub> N(C <sub>3</sub> H <sub>7</sub> O)(CH <sub>3</sub> ) I	5 11.4	97.7	140-3*	11.09 11.21	4.85 4.95	7.52	
<b>Mercaptoethyl Methyl Sulfone</b>								
Mercaptoethyl methyl sulfone	HSC <sub>2</sub> H <sub>4</sub> SO <sub>2</sub> CH <sub>3</sub>	4 13.1	100.6	99-107 (0.1mm.)	45.77 46.40		7.92	
Disulfide				152°C.	46.08 45.97			
a. Lit. 14 1.5015								
b. Lit. 13 1.5033								
c. Lit. 13 1.4795								
d. Lit. 5 n <sub>D</sub> <sup>20</sup> —1.5100								
e. measured at 27°								
f. % I: Calc'd: 46.51; found, 46.49								
*fading end point								

the pyrrolidinium salt, 2 days after initial injection of 0.05 ml. in the same animals caused the death of 6 out of 10 animals, in periods ranging from 1 to 40 minutes. The typical reaction to these injections was early convulsions, usually followed by extreme lassitude before death.

The morpholinium analog was less toxic, but induced transitory tremors and mild convulsions in some of the animals after intradermal injection of 0.10 ml. of 0.17 M. solution. Subcutaneous injection of 0.5 ml. in a 470 gm. guinea pig caused mild convulsions and coma, followed by death in about 20 minutes.

Special reactions were used for a few other compounds. The best preparation of N-mercaptoethyl acetamide was Kuhn and Quadbeck's (5), from ethylene imine:

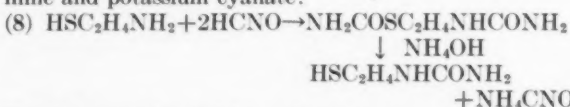


The related compound, N-mercaptoethyl trifluoroacetamide was made from the ester (6) and mercaptoethylamine:



N-mercaptoethylpyrrolidone was prepared by the addition of thioacetic acid to N-vinyl pyrrolidone, and hydrolysis of the addition product.

Mercaptoethyl urea was made from mercaptoethylamine and potassium cyanate:



The beta ureidoethylthiocarbamate precipitated in the cold and was separated, hydrolyzed, and purified.

The mercaptoethylamine derivatives which are prepared are given in Table II.

A related mercaptan, mercaptoethyl methyl sulfone, was made by addition of thioacetic acid to methyl vinyl sulfone (7), and hydrolysis of the addition product. Both

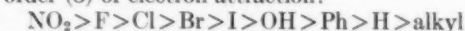
the addition product and the mercaptan were isolated and characterized. The properties were as shown in Table II.

### Disulfides

Disulfides were prepared by oxidation of an aqueous or alcoholic solution of the mercaptan with 5% hydrogen peroxide at 10-15°C. The products were recrystallized and analyzed.

### Variation of Acidity with Structure

The most important variation in properties among the synthesized mercaptans was in the degree of ionization of the sulfhydryl group, which varied from pH 10.2 for thioglycolic acid to pH 6.7 for mercaptosuccinimide. This acidity varied in accordance with the known principle of the inductive effect. In the case of an acid, XCH<sub>2</sub>COOH, increase of the acid strength results when the character of the X group produces an attraction of electrons toward it. Common substituents give the following diminishing order (8) of electron attraction:



Thus, in the case of XCH<sub>2</sub>COOH, the following dissociation constants (8) are noted (Table III):

TABLE III  
Ionization Constants of XCH<sub>2</sub>COOH Compounds

X	Mx10 <sup>-3</sup>	pK
CH <sub>3</sub>	1.34	4.88
H	1.82	4.76
C <sub>6</sub> H <sub>5</sub>	4.9	4.31
OH	15	3.83
SH	28	3.55
Cl	138	2.81
F	220	2.66
C <sub>6</sub> H <sub>5</sub>	8.5	4.07
ClC <sub>2</sub> H <sub>4</sub>	3.0	4.52
ClC <sub>2</sub> H <sub>6</sub>	2.0	4.70

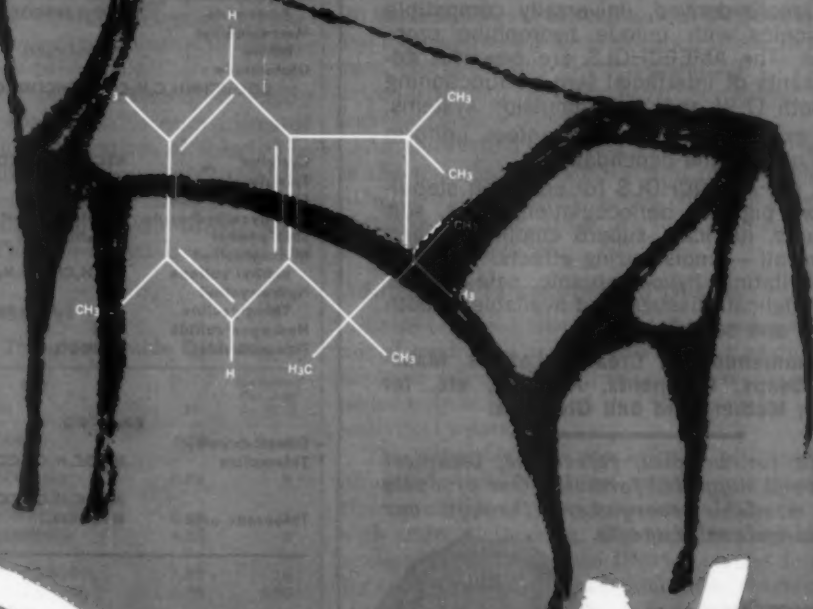
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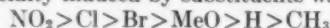
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Table III shows the increase in acidity with increase in negativity of the substituents. The effect is greatly reduced in positions beyond the alpha.

The parallel case of the polarity of the sulfhydryl group (9) will now be considered. At once, the biggest dissociation of the acidic hydrogen is obtained by attaching the group directly to carbonyl in  $\text{CH}_3\text{COSH}$ ,  $K = 4.6 \times 10^{-4}$ . Reid (10) assembled data on substituted thiophenols which bear on the inductive effect. Electron-attracting substituents on the aromatic ring in general increased the sulfhydryl acidity. The order of increasing acidity induced by substituents was:



This is the same order as given above. Other effects in Reid's table are also usual, including the suppression of acidity by parahydroxy (11) and the intensification of acidity by paranitro over metanitro.

The SH group in thioacetic acid has little of the mercaptan character. But this acid, and hydrogen sulfide itself, have been fitted into Table IV, modified from Calvin (9), which shows the variation in acidity of the SH group which results from variation in inductive effects.

**TABLE IV**  
**Dissociation Constants of Mercaptans**

Name	Formula	pK	Dissociation Constant ( $\times 10^{-1}$ )	Reference
Thioglycolic acid	$\text{HSCH}_2\text{COOH}$	10.2	.06	This lab
Ethyl mercaptan	$\text{HSCH}_2\text{CH}_3$	10.0	.1	9
Mercaptoethanol	$\text{HSCH}_2\text{CH}_2\text{OH}$	9.5	.3	9
Thioglycerol	$\text{HSCH}_2\text{CHOHCH}_2\text{OH}$	9.3	.5	This lab
N Mercaptoethyl Acetamide	$\text{HSC}_2\text{H}_4\text{NHCOCH}_3$	8.7	1.5	This lab
Mercaptoethyl Nitrile	$\text{HSC}_2\text{H}_4\text{CN}$	8.6	1.6	This lab
Glutathione	$\text{COOHCHNH}_2\text{C}_2\text{H}_4\text{CONHCHCONHCH}_2\text{COOH}$			
	$\text{CH}_2\text{SH}$	8.7	2	9
Cysteine	$\text{HSCH}_2\text{CHNH}_2\text{COOH}$	8.3	5	9
Thioglycolamide	$\text{HSCH}_2\text{CONH}_2$	8.2	6	This lab
Thioacetic acid	$\text{HSC}_2\text{H}_4\text{COOH}$	8.2	5.8	This lab
Thioglycolhydrazide	$\text{HSCH}_2\text{CONHNH}_2$	8.1	9	This lab
Thio-phenol	$\text{HSC}_6\text{H}_5$	8.0	10	(10)
Mercaptoethyl methyl sulfone	$\text{HSCH}_2\text{CH}_2\text{SO}_2\text{CH}_3$	7.9	12	This lab
Hydroxyethyl Thioglycolate	$\text{HSCH}_2\text{COOCH}_2\text{CH}_2\text{OH}$	7.8	16	This lab
Hydrogen sulfide	$\text{HS}_2$	7.0	91	9
Thiomalimide	$\text{HSC}_2\text{H}_4\text{CONHCO}$	6.7	200	This lab
	$\text{CH}_2 - \text{CO} \begin{matrix} \diagup \text{NH} \end{matrix}$			
Dimethoxyethyl Thiomalate	$\text{MeOC}_2\text{H}_4\text{OOCCHSH}$	6.0	900	This lab
	$\text{MeOC}_2\text{H}_4\text{OOCCH}_2$			
Thioacetic acid	$\text{HSC}_2\text{H}_4\text{COOH}$	4.3	460,000	Int. critical tables

Thus, the acidity of  $\text{H}_2\text{S-X}$  may be varied over a range of a million. Mercaptans have been studied in which the ionization constant of the sulfhydryl was varied from  $10^{-7}$  to  $10^{-11}$ . For the determinations made by us, potentiometric titrations were run by means of the Precision Dow Recordomatic Titrometer from the curves of which the ionization constants of the thiol groups were calculated.

The substitution of OH or CN in  $\text{XCH}_2\text{CH}_2\text{SH}$  has



the expected inductive effect. Other modifications have greater effects. In particular,

- the conversion of COOH to CONH<sub>2</sub> greatly increases the electron attracting effect and makes the mercaptan 100 times more acidic;
- the introduction of S or N in the beta position has a strong effect. Both of these will be discussed below.

#### The Range of Variation of Acidity of the Formula, HSCH<sub>2</sub>COY, where Y Completes an N Derivative or an Ester

The C<sub>2</sub> and C<sub>3</sub> mercaptocarboxylic acids all have about the same ionization constant of the mercaptan group, .06 x 10<sup>-9</sup>. However, for the corresponding amides and hydrazides, the case is quite different.

TABLE V  
Ionization Constants of C<sub>2</sub> and C<sub>3</sub> Mercapto Amides, Esters, and Hydrazides

Radical	Ionization Constants (X 10 <sup>-9</sup> )		
	Amide	Hydrazide	Ester
HSCH <sub>2</sub> CO.....	6.0	9.00	16
CH <sub>2</sub> CHSHCO.....	9.0	—	—
HSCH <sub>2</sub> CH <sub>2</sub> CO.....	0.4	0.07	—

Table V shows the inductive effect of replacement of OH by -NH<sub>2</sub> or -NHNH<sub>2</sub> is far more powerful in the alpha position (cf. Table III). In the ester the mercaptan group is slightly more acidic than in the amide because oxygen has a greater electron withdrawing power than nitrogen. In the preparation of thioglycolamide by passing ammonia into ethanolic thioglycolate ester, it is possible to separate the ammonium salt, NH<sub>4</sub>SCH<sub>2</sub>CONH<sub>2</sub>, by cooling and washing by decantation. This salt yields aqueous solutions of pH 8.5-8.9. After warming in vacuo at 55-60° for three hours to volatilize ammonia the residue gives an aqueous solution of pH 6.0-6.4.

The conversion of acid to amide increased SH acidity one hundred times. The conversion to ester increased SH acidity 250 times. Many derivatives of thioglycolic acid were then found to fall within this range (Table VI). Ways to intensify the acidity further were found, by adding a second activating group to the carbon atom alpha to the mercaptan. These two points are illustrated in Table VI.

TABLE VI  
Ionization Constants of Thioglycolamide Derivatives

Name	Formula	pK	Ionization Constant (x 10 <sup>-9</sup> )
Thioglycolamide	HSCH <sub>2</sub> CONH <sub>2</sub>	8.22	6
N-Methyl Thioglycolamide	HSCH <sub>2</sub> CONHMe	8.05	9
N-Hydroxyethyl Thioglycolamide	HSCH <sub>2</sub> CONHCH <sub>2</sub> CH <sub>2</sub> OH	8.10	8
Thioglycolhydrazide	HSCH <sub>2</sub> CONHNH <sub>2</sub>	8.05	9
Hydroxyethyl thioglycolate	HSCH <sub>2</sub> COOC <sub>2</sub> H <sub>4</sub> OH	7.82	16
Thiomalimide	CHSHCONHCOCH <sub>2</sub>	6.70	200
Dimethoxyethyl thiomalate	CH <sub>2</sub> OC <sub>2</sub> H <sub>4</sub> OOC-CHSH	6.00	900
	CH <sub>2</sub> OC <sub>2</sub> H <sub>4</sub> OOC-CH <sub>2</sub>		

The data in Table VI suggest that the combined effects of two activating groups may be synergistic. It may be seen for example from Table V that the amide group alpha to the sulfhydryl increases acidity by about 10<sup>2</sup> and when in the beta position by about 10, while the imide (both alpha and beta amide type) increases acidity

by a factor of about 3 x 10<sup>3</sup>. In a similar way, the alpha and beta ester groups would be estimated to increase acidity by about 10<sup>3</sup> while the observed effect for the thiomalate diester is 10<sup>4</sup>.

#### The Range of Acidity of the Formula, HSC<sub>2</sub>H<sub>4</sub>NR<sub>1</sub>R<sub>2</sub>

The compounds for which ionization constant was measured, or is available, are given in Table VII.

TABLE VII  
Ionization Constants of Mercaptoethylamine Derivatives

Name	Formula	pK	Ionization Constant (x 10 <sup>-9</sup> )
Mercaptoethyl urea	HSC <sub>2</sub> H <sub>4</sub> NHCONH <sub>2</sub>	9.10	0.8
Mercaptoethyl acetamide	HSC <sub>2</sub> H <sub>4</sub> NHCOCH <sub>3</sub>	8.70	1.5
Mercaptoethyl amine (9)	HSC <sub>2</sub> H <sub>4</sub> NH <sub>2</sub>	8.60	2.5
Cysteine (9)	HSCH <sub>2</sub> CHNH <sub>2</sub> COOH	8.30	5.0
Methyl mercaptoethyl pyrrolidinium iodide	[(HSC <sub>2</sub> H <sub>4</sub> N(C <sub>2</sub> H <sub>5</sub> )(CH <sub>3</sub> )]I	7.77	17
Methyl mercaptoethyl morpholinium iodide	[HSC <sub>2</sub> H <sub>4</sub> N(C <sub>2</sub> H <sub>5</sub> O)(CH <sub>3</sub> )]I	7.49	32
Mercaptoethyl trifluoroacetamide	HSC <sub>2</sub> H <sub>4</sub> NHCOCF <sub>3</sub>	7.28	53

which shows that the introduction of nitrogen in the beta position increases SH acidity over ethyl mercaptan.

Substitution on the amino group of mercaptoethylamine achieved sizeable increase in acidity in two cases. In the first case, the mercaptan was included in a positive ion by quaternizing the amino-mercaptan. In the second case, the powerfully electron-attractive CF<sub>3</sub> group was introduced into mercaptoethyl acetamide. The CF<sub>3</sub> increased acidity 35-fold.

An observation of special interest in these compounds is the remarkable chain distance over which electron-attractive atoms can exert their effect. In mercaptoethyl trifluoroacetamide, there are four atoms in the chain between CF<sub>3</sub> and SH.

#### Addition of Mercaptans to Olefins

Earle (12) said that an aryl thiol such as thiophenol added readily to acrylonitrile in acid medium, but an aliphatic mercaptan such as amyl mercaptan, C<sub>5</sub>H<sub>11</sub>SH, added only in alkali, and that this provided a means for analytical separation.

This suggested that the addition of a mercaptan to an olefinic bond in general would occur more easily as the mercaptan became more acidic. This is in accord with current thinking that addition occurs by the nucleophilic attack of the RS<sup>-</sup> ion on the double bond. By comparing the relevant ionization constants, it was thought that thioglycolamide would add to acrylonitrile in acid medium, but thioglycolic acid only in alkaline. Experimental trial confirmed this. The two mercaptans were treated with acrylonitrile in acid medium. Thioglycolamide added readily, and the nitroprusside reaction rapidly disappeared. But it did not disappear in the case of thioglycolic acid. Reduced hair readily added to acrylonitrile in acid medium, proving the relative acidity of keratin-SH, which is, of course, in accord with acidity of the SH in glutathione.



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### Experimental N-Methylthioglycolamide

A total of 180 g. (1.5 moles) of ethyl thioglycolate was added slowly with stirring and cooling to 111 g. (3.57 moles) of methylamine in 375 ml. of methanol in a 1-litre r.b. flask. On completion of the addition the flask was stoppered and stirred magnetically for one hour. The solution was left at room temperature overnight. The methanol was then distilled at atmospheric pressure and the residue was subjected to a vacuum distillation. A total of 123.3 g. of a water-white liquid was obtained, distilling at 91—4° at 5 mm. pressure. % conversion—78.3. Purity by thiol titration—99.8%. Calculated for  $C_3H_7NOS$ : N, 13.3; S, 30.5. Found: N, 12.9; S, 30.6.

The N,N-dimethyl, N-ethyl, N-isopropyl, N-amyl, N-hexyl, N-heptyl, and N-decyl derivatives were prepared in an analogous fashion, except that a considerably longer reaction time was necessary, especially for the longer chain compounds. Furthermore, the reagents were combined instantly and the flask was stoppered and stored at temperatures up to 140°F and times up to four weeks (in the case of the N-decyl derivative). The extent of completion of the reaction was determined by titration of a sample of the reaction mixture with standard hydrochloric acid using methyl red indicator.

The N-hydroxyethyl, N-methoxyethyl, N-glyceryl, N-(diethylaminoethyl) derivatives and thioglycolhydrazide were prepared in a similar manner to the N-alkyl derivatives. In the syntheses of the first four compounds, the ester and the amines were combined in the absence of solvent. A polyethylene glycol analog was made from Dow polyglycolamine 175.

### 2-Mercaptopropionamide

A total of 48.0 g. (0.40 moles) of methyl 2-mercaptopropionate was added to a solution of 14.5 g. (0.85 moles) of ammonia in 170 ml. of methanol in a 500 ml. r.b. flask. The flask was stoppered and set aside at room temperature for one week. At the end of this time the volatiles were removed under vacuum, the residue finally being heated to 60° for two hours. Upon cooling, there was obtained 37.0 g. of a white, crystalline solid. % Conversion—88.0. Purity by thiol titration—93.2%.

### 2-Mercaptopropiohydrazide

A total of 15.0 g. (0.30 moles) of hydrazine hydrate was added to 44.7 g. (0.272 moles) of methoxyethyl 2-mercaptopropionate dissolved in 100 ml. of methanol in a 500 ml. r.b. flask. The flask was tightly stoppered and stored at room temperature for one week. The volatiles were removed under the aspirator, the product finally being heated to 120°C for two hours. On cooling, a white solid was obtained in a conversion of 93.4%. Purity by thiol titration—88.3%. Disulfide content—5.0%.

### 3-Mercaptopropionamide

7.1 ml. of thioacetic acid and 7.1 g. of acrylamide were mixed. Addition occurred with strong evolution of heat. The reaction product solidified on cooling. 13.7 g. was broken up and mixed with 15.3 g. of concentrated ammonia. After standing overnight, iodine titration showed 91% of the theoretical mercaptan.

### 3-Mercaptopropiohydrazide

This preparation was similar to that of the 2-mercaptopropionamide.

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compound. Purity by thiol titration—96.5%. Disulfide content—1.1%.

### N-Mercaptoacetyl Glycine

Acetylmercaptoacetyl chloride (0.2 mol), from the reaction of the acid with thionyl chloride; and ethyl glycinate (0.2 mol), were added at the same rate from two dropping funnels into a well-stirred suspension of 87 grams  $\text{CaSO}_4$  and 100 grams  $\text{K}_2\text{CO}_3$  in 400 ml. ethyl ether, at 10-15°C. Stirring was continued for 3 hr. at room temperature. The mixture was filtered, and the filter cake washed with ether. The product, ethyl acetyl mercaptoacetyl glycerine, was crystallized from the ether solution. M.p. 78.5°C. Calculated C 43.85, H 5.94, N 6.39, S 14.61; found C 43.95, H 5.91, N 6.40, S 14.53.

7.1 grams (.032 mol) of this product was treated with .09 mols of ammonia in aqueous solution to form the mercaptan. The solution titrated .032 mols (100%) of the mercaptan with iodine.

### N-Mercaptoacetyl Glucosamine

Tetra-acetyl glucosamine was prepared by acetylation of the p-methoxybenzylidene-protected amine. The regenerated tetra-acetyl amine (0.17 mol) was dissolved in 300 ml chloroform. At 0°C, with stirring, 0.08 mol of acetyl mercaptoacetyl chloride in 38 ml chloroform was added. When a precipitate formed, 550 ml chloroform was added. Stirring was continued 3 hours. The mix was filtered, and the filter-cake washed with chloroform. The filtrate was concentrated in vacuo and diluted with petroleum ether to precipitate the product, N-acetylmercaptoacetyl tetra-acetyl glucosamine. Yield 77%, m.p. 190-191°C. Calculated C 46.64, H 5.44, N 3.02, S 6.92. Found C 46.89, H 5.56, N 3.21, S 7.09. Product, dissolved in alcohol and precipitated with a little water, gave N 3.00, S 6.96.

15.1 grams (.033) mol of the product was treated with 30.1 ml  $\text{H}_2\text{O}$  and 13.8 gram concentrated ammonia (0.23 mol  $\text{NH}_3$ ) to form the mercaptan. After 24 hours, the solution titrated .013 mols (39%) of the mercaptan with iodine. A few days later, 1.5 grams solid  $\text{NaOH}$  was added. After 24 hours, the solution then titrated 0.23 mol (69%) of the mercaptan.

### N-Mercaptoacetyl Aminomalonic Acid

Acetylmercaptoacetyl chloride (0.59 mol), and diethyl aminomalonnate (0.55 mol), from the catalytic reduction of nitromalonic ester, were added as 5% ethereal solutions at the same rate from two dropping funnels into a well-stirred suspension of 187 gram  $\text{CaSO}_4$  and 215 gram  $\text{K}_2\text{CO}_3$  in 860 ml ethyl ether, at 5-10°C. Stirring was continued 2 hours with cooling and 1 hour at 10-25°C. The mixture was filtered, and the filter cake washed with ether. The product, diethyl N-acetylmercaptoacetyl aminomalonnate, was crystallized from the ether solution. Yield 52%, m.p. 91.5°C. Calculated N 4.87, S 11.22; found N 4.81, S 11.0.

10.0 grams (.034 mol) of this product was treated with .15 mols of ammonia in aqueous solution to form the mercaptan. The solution titrated .035 mols (103%) of the mercaptan with iodine.

### Thioglycolamide Derivatives

By reaction of the appropriate alcohols with ethyl



thioglycolate in sealed tubes at 140°F. for 48 hours, and removal of volatiles, two thioglycolamide derivatives were obtained as syrupy liquids of 85-88% purity by iodine titration. These compounds were  $\text{HSCH}_2\text{CON}(\text{C}_2\text{H}_4\text{OH})_2$  and  $\text{HSCH}_2\text{CONHC}(\text{Me})(\text{CH}_2\text{OH})_2$ . Reaction of thioglycolate with the related alcohol,  $(\text{HOCH}_2)_3\text{CNH}_2$ , to produce  $\text{HSCH}_2\text{CONHC}(\text{CH}_2\text{OH})_3$ , required 16 days storage of the stoichiometric mix in a sealed tube at 140°F with occasional shaking. The yield by thiol titration was 66%.

Beta mercapto propionethanolamide,  $\text{HSC}_2\text{H}_4\text{-CONHC}_2\text{H}_4\text{OH}$ , was prepared by adding thioacetic acid to methylacrylate and mixing the resultant addition product with ethanolamine. Two moles of amine were used, the extra one to hydrolyze the S-acetyl group when the product was dissolved in water. The yield by thiol titration was 88%.

Mercapto succinimide was prepared by the addition of thioacetic acid to maleimide in the presence of benzoyl peroxide. The liquid addition product soon crystallized, m.p. 91°C. Treatment with methanolic hydrogen chloride and removal of volatiles left a syrupy liquid, thiol purity 89%,  $\text{pK} = 6.65$ .

#### N-Mercaptoethylmorpholine

A total (13) of 113 g. (1.30 moles) of distilled morpholine and 78 g. (1.30 moles) of ethylene sulfide was combined in a 400 ml constricted tube, which was then cooled and sealed (14). The tube was surrounded by acetone in an autoclave and held at 100° for sixteen hours. The tube was opened and the contents were rectified through a 5' Podbielniak column under vacuum. There were 149 g. of product distilling at 72-3° at 0.9 mm. % Conversion—77.9. Thiol purity—98.8. The other constants and analyses are shown in Table II.

#### N-Mercaptoethylpyrrolidine

A 1-litre 3-neck flask was fitted with a sealed stirrer, dropping funnel and condenser whose outlet was connected through a drying tube to a tower of Fieser's solution. A total of 68.5 g. (1.14 moles) of ethylene sulfide was added dropwise with stirring to a solution of 81 g. (1.14 moles) of distilled pyrrolidine in 400 ml of dry dioxane. On completion of the addition, during which moderate warming occurred, the solution was refluxed with stirring for six hours. The dioxane was distilled at atmospheric pressure and the residue was rectified through the 5' Podbielniak column. There were obtained 111 g. of water-white distillate boiling at 78-80° at 20 mm. Hg. % Conversion—74.2. Thiol purity—96.5%. The other constants and analyses are shown in Table II.

The remaining mercapto amines, N-mercaptoethylpiperidine, 3-aza-6-oxaheptanethiol, and 3-aza-5-diethylaminopentanethiol were prepared in essentially the same manner as N-mercaptoethylpyrrolidine. Their physical constants and analyses are shown in Table II.

#### N-Mercaptoethyl-N-Methylpyrrolidinium Iodide

A—N-(S-Acetylmercaptoethyl) pyrrolidinium Chloride. A 2-l. 3-neck r.b. flask was fitted with a sealed stirrer, thermometer, condenser and dropping funnel, the latter two units being equipped with drying tubes. A solution of 39.3 g (0.5 moles) of acetyl chloride in 400 ml of benzene was added dropwise with stirring to a solution of 65.5 g (0.5 moles) of N-mercaptoethylpyrrolidine in

300 ml of benzene, the temperature being maintained at 20-30°. On completion of the addition, the mixture was refluxed for ten minutes. Upon cooling, 400 ml of hexane was added and the precipitate was filtered off. It was washed with 200 ml of a 50-50 benzene-hexane mixture and placed in a vacuum desiccator over paraffin. Weight—85.5 g. % Conversion—81.6. M.p. 152°. Calculated for  $\text{C}_8\text{H}_{16}\text{ClNOS}$ : Cl, 16.94; N, 6.68; S, 15.30; found: Cl, 16.81; N, 6.65; S, 15.34.

B—N-(S-Acetylmercaptoethyl) pyrrolidine.

A total of 52.5 g (0.25 moles) of N-(S-acetylmercaptoethyl) pyrrolidinium chloride was slurried in 25 ml of water in a 500 ml 3-neck flask equipped with thermometer, stirrer and dropping funnel. There was added 200 ml of ether and the mixture was cooled to 0-5°. With vigorous stirring 400 ml of 30% sodium hydroxide was slowly added, the temperature being maintained at 0-5°. On completion of the addition the aqueous layer was saturated with anhydrous potassium carbonate and the ether layer was separated. The aqueous layer was extracted with three 150 ml portions of ether and the combined extracts were dried for one hour over potassium carbonate. After removal of the volatile, a vacuum distillation yielded 34 g of water-white liquid boiling at 65° at 0.2 mm. % Conversion—78.5.  $N_{20} = 1.5011$ . Cal-

D  
culated for  $\text{C}_8\text{H}_{16}\text{NOS}$ : N, 8.08; S, 18.52. Found: N, 8.01; S, 18.31.

C—N-(S-Acetylmercaptoethyl)-N-methylpyrrolidinium Iodide.

A total of 34 g. (0.24 moles) of methyl iodide was added to a solution of 29 g. (0.168 moles) of N-(S-acetylmercaptoethyl) pyrrolidine in 300 ml of anhydrous ether. The solution was stoppered and stored eight days at room temperature. At the end of this time the voluminous precipitate was filtered off and recrystallized from ethanol. Weight—45.0 g. % Conversion—85.0. M.p. 159-60°. Calculated for  $\text{C}_9\text{H}_{18}\text{INOS}$ : N, 4.44; S, 10.18. Found: N, 4.36; S, 10.18.

D—N-Mercaptoethyl-N-methylpyrrolidinium Iodide.

A total of 43.0 g. (0.156 moles) of N-(S-acetylmercaptoethyl)-N-methylpyrrolidinium iodide was dissolved in 460 ml of 0.22 N methanolic hydrogen chloride in a 500 ml flask, which was then tightly stoppered. This solution was stored six days at room temperature after which a thiol titration indicated conversion to the mercaptan to be 84.0% complete. The volatiles were removed under vacuum to leave a slightly moist, white, crystalline residue. This crude product (28.8 g.) was recrystallized from ethanol with a minimum exposure to air. Weight—19.0 g. % Conversion—44.8. M.p. 66-7°. Thiol purity—99.7%. Calculated for  $\text{C}_7\text{H}_{14}\text{INS}$ : I, 46.51; N, 5.13; S, 11.75. Found: I, 46.49; N, 5.15; S, 11.81.

#### N-Mercaptoethyl-N-Methylmorpholinum Iodide

The route to N-mercaptoethyl-N-methylmorpholinum iodide was completely analogous to the above, the starting material in this case being N-mercaptoethylmorpholine. The data on the intermediates were as follows:

A—N-(S-Acetylmercaptoethyl morpholinum Chloride—% Conversion—92.2. M.p. 204°. Calculated for  $\text{C}_8\text{H}_{16}\text{ClNOS}$ : N, 6.21; S, 14.22. Found: N, 6.11; S, 14.43.

B—N-(S-Acetylmercaptoethyl) morpholine—% Conversion—59.5. B.p. 87° at 1.5 mm. Hg.  $N_{20} = 1.5050$ . No analyses.  
D

C—N-(S-Acetylmercaptoethyl)-N-methylmorpholinium Iodide—% Conversion—85.5. M.p. 136-9°. Calculated for  $C_{10}H_{18}INO_2S$ : I, 38.34; N, 4.23; S, 9.68. Found: I, 38.00; N, 4.17; S, 9.57.

The starting materials were refluxed in benzene to prepare this compound.

D—N-Mercaptoethyl-N-methylmorpholinium Iodide—% Conversion—11.4. M.p. 140-3°. Thiol purity—97.7%. Calculated for  $C_7H_{14}INOS$ : I, 43.92; N, 4.85; S, 11.09. Found: I, 40.23; N, 4.95; S, 11.21.

### N-Mercaptoethyltrifluoroacetamide

A solution of 2 g. (0.087 moles) of sodium in 50 ml of anhydrous methanol was placed in a constricted Pyrex tube. This was thoroughly shaken with 10.5 g. (0.092 moles) of mercaptoethylamine hydrochloride and 28 ml of ethyl trifluoroacetate was added. The tube was sealed and heated at 100° for 16 hours in a rocking autoclave containing methanol to equalize the pressure. Upon opening the tube, the contents were filtered and the volatiles were removed. A vacuum distillation yielded 11.5 g. of water-white liquid distilling at 78.85° at 1-2.5 mm. Hg. Thiol purity—59.8%. An I.R. absorption spectrum indicated the presence of the expected functional groups and gave no indication as to what impurities might be present. The product was rectified through the 5' Podbielniak column and yielded approximately 6 g. of distillate possessing a thiol purity of 70%.

The entire preparation was repeated, this time using a slight excess of sodium methoxide. There was obtained 4 g. of product possessing a thiol purity of 88.8%.

The two products were combined and rectified through the spinning band micro Podbielniak column. There were obtained three cuts as follows:

Cut No.	Volume	Temperature	Pressure	Thiol purity
1	2.05 ml	57-61°	1.2 mm	98.5%
2	4.25 ml	61-2°	1.2 mm	81.1%
3	0.60 ml	62-5°	1.2 mm	—

Complete data and analyses were obtained on cut no. 1 which appeared to be of the highest purity. % Conversion—9.1. Calculated for  $C_4H_8F_3NOS$ : F, 32.9; N, 8.09; S, 18.53. Found: F, not more than 35.23; N, 8.46; S, 19.38.

### N-Mercaptoethylpyrrolidone

N-Mercaptoethyl pyrrolidone was prepared by the addition of thioacetic acid to N-vinyl pyrrolidone. The mercaptan was liberated from the light yellow, non-viscous liquid additive product by dissolving in aqueous ammonia. It was not isolated, but studied in solution. The addition product crystallized on standing, m.p. 37°C. For  $(C_4H_6ON)C_2H_5SCOCH_3$ , calculated (%) S, 17.1; N, 7.48; C, 51.4; H, 6.95. Found: S, 17.5; N, 7.16; C, 51.0; H, 6.77.

### Mercaptoethylurea

To an aqueous solution of 0.5 mol of mercaptoethylamine hydrochloride was added 1.0 mole of potassium cyanate, and then concentrated HCl. The temperature was kept at 15-20°C. When reaction ceased, the mixture was chilled overnight. The separated beta ureidoethylthiocarbamate was warmed in a beaker with two equivalents of ammonium hydroxide until all dissolved. A crude product was separated by concentration in vacuo, and purified by Soxhlet extraction with chloroform. The

product separating from chloroform solution titrated 97% of the theoretical thiol. Ionization constant,  $0.8 \times 10^{-9}$ . The disulfide of this mercaptan has relatively very low solubility in water.

### Mercaptoethyl Methyl Sulfone

A—S-Acetylmercaptoethyl methyl sulfone.

A total of 61 g. (0.58 moles) of methyl vinyl sulfone was refluxed gently with 48 g. (0.63 moles) of thioacetic acid and a crystal of benzoyl peroxide, a drying tube being connected to the condenser outlet. After 20 hours the mixture became a dark brown semi-solid mass. This was treated with 400 ml of boiling ethanol and the mixture filtered. The filtrate, upon cooling yielded a dark brown solid which was filtered. After removal of the volatiles from this filtrate, the residue was vacuum-distilled. It yielded 35 g. of a reddish-brown liquid distilling at 124-38° at 0.3 mm Hg. This solidified on cooling and was recrystallized twice from absolute ethanol with addition of Nuchar. There was obtained 16.0 g. of pale yellow crystalline melting at 59-60°. % Conversion—15.2. Calculated for  $C_5H_{10}O_2S_2$ : S, 35.22. Found: S, 35.89.

B—Mercaptoethyl methyl sulfone.

A total of 19.7 g. (0.108 moles) of S-acetylmercaptoethyl methyl sulfone was dissolved in 475 ml of a solution of 0.22 N methanolic hydrogen chloride. This solution was tightly stoppered and stored five days at room temperature, after which a thiol titration of an aliquot indicated conversion to the mercaptan to be quantitative. The volatiles were removed and the residue was subjected to a vacuum distillation. This yielded 13.0 g. of a pale yellow liquid distilling at 99-107° at 0.1 mm Hg, most distilling at 105-7°. % Conversion—85.9. Thiol purity—100.6% N<sup>20</sup>—1.5240. d<sub>4</sub><sup>20</sup>—1.3208. Calculated for  $C_3H_6S_2O_2$ : S,

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45.77; Found: S, 46.40.

### Hydroxyethyl Thioglycolate

This ester was prepared by removal of water from a thioglycolic acid—ethylene glycol mixture (1:1.1) using a Dean-Stark tube and p-toluene sulfonic acid catalyst. B.P. 86-91°C. at 20-30 mm. pressure. Thiol purity, 99.4%.

### Dimethoxyethyl Thiomalate

Water was removed from a thiomalic acid—methoxyethyl alcohol mixture (1:2) using a Dean-Stark tube and p-toluene sulfonic acid catalyst. B.P. 130-134°C. at 0.5 mm. Thiol purity 99.2%.

### Acknowledgements

Acknowledgements for portions of the synthetic work are due to Dr. J. E. Callen, to Dr. Murray Berdick of Evans Research and Development Corporation, and to Dr. W. E. Taft, now of American Cyanamid Company.

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#### DOROTHY GRAY—1

Dorothy Gray has demonstrated how packaging costs can be kept down when the products are marketed abroad. Through the International Division of Lehn & Fink Products Corp., both Better Off depilatory, shown here, and Sheer Velvet Hair Spray are sold widely in Latin American Markets. Print runs with alternate Spanish labeling and instructions for the depilatory are tied in with domestic runs and plates simply substituted. For the hair spray, instructions are printed in both English and Spanish. This is marketed throughout Latin America and in some other foreign markets.

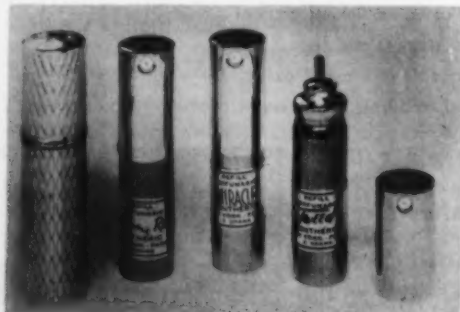


#### LENTHERIC—2

"Perfumagic," Lentheric's new perfume package, is believed to mark the first time a fragrance house has promoted the refillable possibilities of the new purse-size aerosol containers.

The decorative "Perfumagic" case, made of mat-silver with a slender gold thread design, can be purchased with an inner cartridge holding one of the three Lentheric scents—Tweed, Adam's Rib or Miracle. When the original fragrance is used up, the inner container can be removed easily and a new Lentheric fragrance inserted in its place. Or the customer can buy refills when she purchases the "Perfumagic" and interchange them as frequently as she wants.

The refills click in and out as easily as do the popular replaceable lipsticks.



#### YARDLEY—3

The first Yardley of London promotion of 1960 features Lavender soap, presented in a special pack of four tablets for \$1.50, the regular retail price of three. In limited supply, the dividend pack will be available in leading department and drug stores around February 15. To call consumer attention to the free offer, the four-tablet box features an end sleeve in red imprinted with the legend, "One Tablet Free with Every Three." The sleeve is easily removable, leaving the box suitable for a gift. A self-service counter display unit holding one dozen dividend packs is available to retailers. Topping the display unit is a placard bearing the message, "Get The Quality Habit and Save Too!"





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#### HELENA RUBINSTEIN—4

Young Touch Hand Lotion by Helena Rubinstein, shown here in a new glass bottle ringed and lettered in a deep shade of turquoise blue, is a rich, hand smoothing lotion made with Estrogenic Hormones. Its purpose: to help prevent discolorations and age signs on the hands while it works against roughness.

#### RICHARD HUDNUT

A new Hand Lotion—in an introductory two-bottle combination has been announced by Richard Hudnut. The new Hand Lotion contains enzymes and its exclusive Alginate Formula leaves the hands and body smooth and soft, conditions the skin to prevent winter chapping, prevents detergent burn. This new formulation is non sticky, leaves no film and is quick drying. The Richard Hudnut Hand Lotion comes packaged in a boot—offering two sleek-form \$1.00 bottles holding 5 ounces each at a special introductory price of \$1.50 plus tax.

#### COTY—6

Aimed at additional holiday gift volume this Valentine store display unit features items in COTY'S most popular fragrance—L'AIMANT. In prices ranging from \$2.50-\$5.00 it includes perfume, toilet water, spray mist and dusting powder. Another important feature of this unique and compact display is its easily convertible to year-'round use when you slip the heart decorations from the packages and display unit. L'AIMANT is supported all year by extensive national television over the ABC network—Beat The Clock with Bud Collyer, The Gale Storm Show and Dick Clark's American Bandstand. There is also a heavy schedule of TV Spots in key markets and a campaign in major women's magazines—Ladies' Home Journal, Glamour and Seventeen.

#### JULIETTE MARGLEN—7

Juliette Marglen has introduced Cellini Gold, Silver and Bronze makeup for lips, fingernails and eyes. The newest of these is Eye Colour, which is reportedly waterproof, smear-proof and non-irritating. For the lips, the Juliette Marglen Oval Lipstick has two new shades, Cellini Bronze, a glittering gold-fired burnished red, and Cellini Gold. For the nails there is Juliette Marglen's metallic spangle—Cellini Gold, Silver and Bronze Nail Glacé.



6.



7.

## C.S.M.A. ABSTRACTS

(Continued from page 38)

### PROGRESS OF LEGISLATION ON LABELING HAZARDOUS CHEMICAL

F. Dallas Sparre,  
E. I. DuPont de Nemours & Co., Inc.,  
Wilmington, Delaware

A brief review of the jurisdictions currently requiring precautionary labeling of hazardous chemicals with particular emphasis on those areas which have laws based on the model bill agreed to by Chemical Specialties Manufacturers Association. Mention is made of new areas most likely to pass such legislation. Specific labeling requirements and preparation of labels are not discussed.

### BACILLUS THURINGIENSIS, BERLONER, AS AN INSECTICIDE

James M. Merritt and M. A. Manzelli,  
Merck & Company, Inc., Chemical Division,  
Rahway, New Jersey

The classical popular interest in biological control of insects is supporting a significant developmental program. The long-known pathological effects of *Bacillus thuringiensis* spore preparations are being applied as insecticidal sprays and dusts supplied by fermentation process industry production. The knowledge that has been thus accumulated during the past two seasons is defining a useful new class of commercial insecticides. New aspects of microbiology are developing, along with more details of insect physiology and toxicology. The future holds great promise for insect control. However, perplexing problems are developing for the supplier industry unless new concepts of cooperative developmental action are evolved.

### DISTRIBUTION AND SHIPPING

William F. Dahms and Charles J. Strom,  
R. M. Hollingshead Corporation,  
Camden, New Jersey

The importance of the time element. The effect of Modern Materials Handling in Shipping & Distribution. Observing of rules and regulations issued by the Interstate Commerce Commission on Shipments. How a chemical manufacturer handles his shipping and distribution problems. The proper classification and routing of shipments. The utilization of space in the shipping department to make for more efficiency and less handling. The part played by the Traffic Department in the overall transportation and its importance to top management in the company. The proper handling of paperwork and importance of accuracy. What efficient distribution means to the very existence of any company. The consolidation of shipments for savings in freight. To keep up with modern trends in material handling for constantly seeking ways to improve and advance your company.

## DEVELOPMENTS IN CONTAINERS

(Continued from page 23)

migrate, and the possibility of exterior entrapment is possible, this potentiality should be considered.

**9. Container Inertness for Food and Drugs**—All manufacturers of packaging materials for the food industry are very much aware, at this time, of the problems involved with the new Federal "Additives Amendments." This requires that there must be no transfer of the packaging materials or containers to the contained foodstuffs, either by extraction, by mechanical removal or any other process. In effect, this calls for careful evaluation of the specific material that may be employed for a specific product and calls for extensive evaluation of not only the parent materials employed, but also the plasticizers, colorants, stabilizers and pigments used. In a recent Packaging Institute Forum, Ramsey (4) stated, "Of the basic packaging materials, wood, glass and recognized metals or alloys are unlikely to present any unresolved questions of suitability because of their long history of satisfactory usage." He further emphasizes the need for careful laboratory evaluation of extraction rates of plastic containers for each specific food to be packaged.

**10. Light Transmission Characteristics**—In many instances, it is ideal to have containers which are transparent so that the products may be viewed, as with flint glass. On the other hand, most of semi-rigid plastic materials are opaque to translucent with only several of them transparent. Further, if desired, it must be possible to provide a screening of certain ranges of light to prevent photochemical changes of the contents. The U. S. Pharmacopoeia (5) recognizes that wave lengths from 290-450 mμ are very active photochemically, and that where the products are sensitive, definite screening is advisable. This may be accommodated by such arrangements as amber glass, opaque containers or the addition of specific screening agents to plastic materials. However, the addition of screening agents must be checked for possible migration to the product.

In summary, each of the enumerated ten factors must be evaluated by the potential user of rigid or semi-rigid containers, predicting in each instance the potential effect, if any, on the contained product, and the reasonable shelf life. The economic price range for each container, particularly including all secondary packaging, must then be weighed against the potential advantages of the specific container, its mechanical or physical characteristics, and its relative ability to approach the "ideal container".

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COSMETIC  
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CHEMICALS

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ACTAMAR  
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AMMONIATED  
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CASTOR OIL  
& SULPHONATE  
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COLORS  
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GLYCERYL ESTERS  
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HEXYLENE GLYCOL  
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ALCOHOL 99%  
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MYRISTATE

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PALMITATE  
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KAOLIN COLLOI-  
DAL  
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KARAYA GUM  
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LACTIC ACID 85%  
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LANOLIN  
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MENTHOL  
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MERCURIC SUL-  
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METHYL CELLU-  
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MINERAL OIL  
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PHENOL CRYSTALS  
OR LIQUID

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CURIC NITRATE  
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PILOCARPINE  
HYDROCHLORIDE  
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POLYETHYLENE  
GLYCOLS & ESTERS  
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POTASSIUM  
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PROPHENPYRID-  
AMINE MALEATE  
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PROPIONIC ACID  
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QUINCE SEED  
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SULFOSUCCINATE  
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SODIUM LAURYL  
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## PRODUCTS & IDEAS



### Fluorine Analyzer

A new fluorine analyzer of liquids and solids employing Nuclear Magnetic Resonance (NMR) techniques is announced by Ridgefield Instrumentation Division, Schlumberger Well Surveying Corporation.

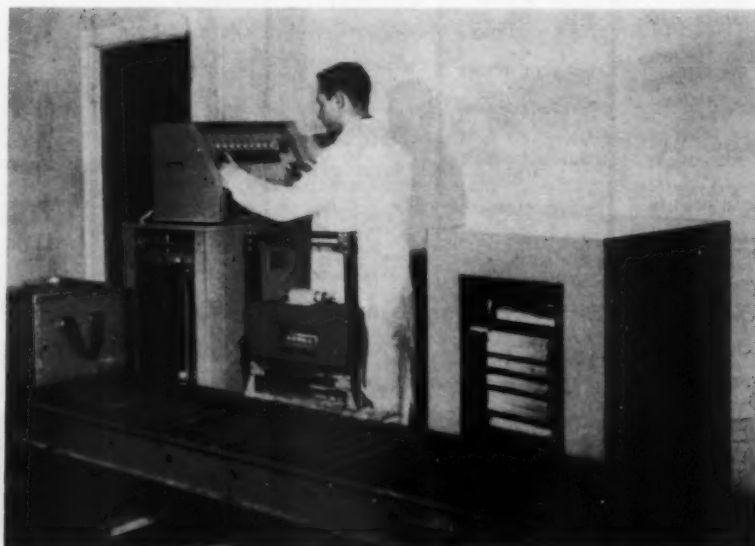
Designated the Model 105 Analyzer, the instrument can detect fluorine in liquids down to 3 mgm in any sample size from 0.2 to 40cc. When the smallest sample size is used, the higher field homogeneity permits high-resolution structure analysis to be applied. Fluorine peaks which are chemically shifted more than 9 ppm can be cleanly resolved. An integrator accessory enables the operator to obtain a linear response, in terms of the mass of fluorine present. When chemical shift is great enough, each peak in an unknown mixture can be separately integrated or, with equal ease, the mass of fluorine present can be read out. Schlumberger Well Surveying Corp.

### Fluorine Analyzer ▲

### ▼ Automatic Routing

### Automatic Routing

A new automatic conveyor routing system has been announced by Atronic. The Model 450 System automatically directs tote boxes, cardboard cartons and other containers to selected stations on a conveyor line. The automatic routing system can be used to direct containers to packing stations for broken lot order assembly; or to direct work through various processing stations. Extreme flexibility permits either single containers or "trains" of containers to be handled. It is specially designed so that the container can be diverted at any station, processed, placed back on the conveyor and diverted again at another station for further processing. The routing system is controlled by a dispatcher at the control console. He selects the routes by depressing station keys and the coded instructions are automatically placed on the container by an inking printer. Code reading Carton Selectors accurately control the direction of flow.



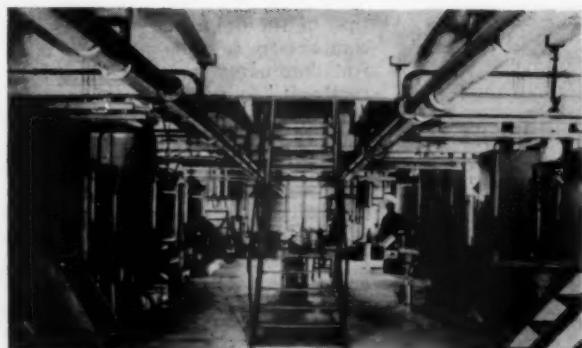




# CAPACITY



# QUALITY

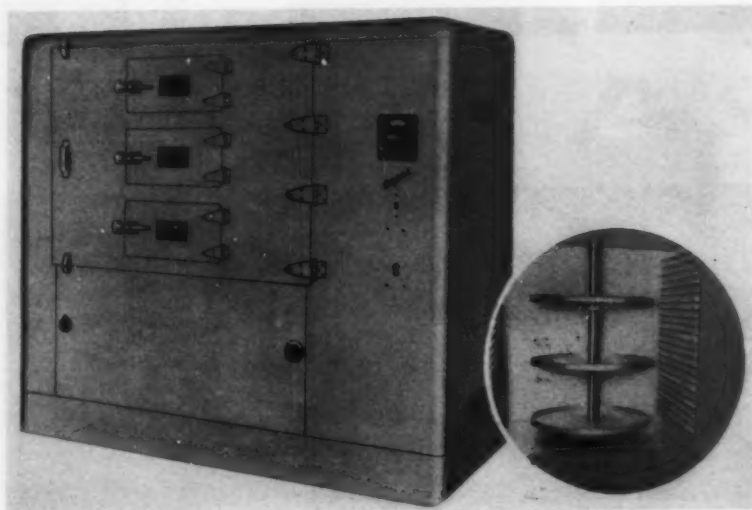


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## PRODUCTS & IDEAS



Laboratory Oven ▲

Duo-Spectranal ▼



### Laboratory Oven

The new Despatch rotary shelf laboratory oven has rotary shelves, either manually or mechanically operated that are spaced on 12 inch vertical centers. The maker recommends manual operation for temperatures below 300° F. For higher temperatures to the oven maximum of 850° F., there is a motor and reduction gear provided to maintain constant shelf rotation. The rotary shelf oven comes in all sizes of Despatch "V" oven series. This series can be supplied for gas, electric or steam heat. It utilizes the Despatch forced convection with horizontal air flow. Even without the rotary shelf this model maintains heat uniformity within  $\pm 1^\circ \text{C}$ . throughout the work chamber. Other features of this series include hinged plug-type doors located within one main door on same level as rotary shelf for easy removal of product. Each door is provided with heat resistant glass window. Despatch Oven Co.

### Fisher Duo-Spectranal

Fisher Scientific Co. has introduced a portable spectroscope which, according to the company, can not only supply a quick qualitative analysis for the common metals and a number of other elements but can be used for semi-quantitative analysis as well. The Duo-Spectranal shows the operator two spectra—that of the unknown, and that of a comparison solution—side by side. An illuminated scale, graduated directly in Angstrom units, can be projected beside the spectra and the wavelength of any line read to  $\pm 5\text{\AA}$ . The two sources—unknown and comparison sample—are automatically aligned when the sample stand is slipped in place. Each sample tube is enclosed in a water-jacket and sealed by a Lucite plug holding a pair of precisely-positioned platinum electrodes. (Most samples are dissolved in 10% nitric acid solution.)

The spectroscope slit "sees" both sets of electrodes. The two beams of light from the slit are focused on the grating and dispersed into two spectra, located side-by-side in the field of a Ramsden-type eyepiece. A neoprene eyeshield insures accurate comparisons, even in a lighted room.

By turning a scanning knob on the instrument, the operator sweeps spectra and scale across the field of the eyepiece, studies any section. Fisher Scientific Co.

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# Trade Literature

## Alkanolamides

● New technical literature describing the properties and uses of Alkanolamides and Glycol Esters is now available from the Clintwood Chemical Company, 33 North LaSalle Street, Chicago 2, Illinois. The functions of the alkanolamides as foam boosters, foam stabilizers, thickeners, detergents and emulsifiers are described. Formulas for shampoos, liquid detergents for "all purpose" household cleaners, dishwashing compounds and floor cleaners are included. Directions for the use of opacifying agents in cosmetic preparations and liquid detergents are given in detail.

## Carbide Booklet

● A new 20-page booklet, describing the properties and uses of pyridines and piperazines, has been published by Union Carbide Chemicals Company, Division of Union Carbide Corporation. The booklet contains extensive data on five pyridines and six piperazines, including physical properties, constant-boiling mixtures, solubilities, shipping data, storage and handling, specification limits, test methods and physiological properties. It also includes a detailed bibliography and reference section. The pyridines have been of particular interest in the manufacture of pharmaceuticals since the structure appears so frequently in the chemistry of living matter. Increased availability from petrochemical sources has encouraged other uses, including surfactants, monomers, and chemical intermediates.

## Reilly Chemical Index

● The fifth edition of the Reilly Chemical Index has just been published by Reilly Tar & Chemical Corporation. It contains a comprehensive, but not complete, listing of the chemicals produced by Reilly, listed alphabetically and according to classes. A copy of the new Reilly Chemical Index may be obtained from Reilly Tar & Chemical Corporation, Merchants Bank Building, Indianapolis 4, Ind.

## "Organic Structures" Type

● How to type the chemical Benzene Ring in a matter of seconds with Remington Rand Interchangeable Type Heads is the subject of a new folder just published by Remington Rand Division of Sperry Rand Corporation. Much data in chemistry cannot be expressed accurately without the use of geometric patterns called "organic structures"—series of hexagonal designs expressing chemical relationships. The Benzene Ring is perhaps their most common form and might appear in a number of different ways. With the new Remington Electric Typewriter—equipped with just two

interchangeable type bars—the typist merely snaps two special Heads into the bars and she is ready to type the Benzene Ring. A copy of this folder can be obtained at any Remington Rand branch office, or by writing to the company at 315 Park Avenue South, New York 10, N. Y. and requesting RE 9027.

## Control Bulletin

● A 4-page bulletin describing their new Electronic Solution Controller is available from Electro Mechanisms Corp., 7301 Ridgeway Ave., Skokie, Ill. Material describes automatic control of concentration of caustic, acidic or aqueous solutions in either continuous or batch-type metal washing equipment, plating processes, chemical process systems, textile bath equipment, and other applications where precise concentrate levels must be maintained. Also described are system components: Sensing Electrode, Magnetic Amplifier, Pump Assembly, Wash Tank and Holding Tank.

## Physical Properties Booklet

● A new 28-page Physical Properties booklet, describing more than 400 synthetic organic chemicals, has been published by Union Carbide Chemicals Company, Division of Union Carbide Corporation. The booklet contains information on applications, physical properties, and shipping data. Included are alcohols; acids; anhydrides; chlorine compounds; esters; ethers and oxides; glycol-ethers; glycols and triols; ketones; monomers; Carbowax polyethylene glycols; metallic salts; nitrogen compounds; cyclic compounds; Flexol plasticizers; polypropylene glycols; Ucon fluids, lubricants, and fluorocarbons; Tergitol surfactants; Polyox water-soluble resins; Cellosize hydroxyethyl cellulose; new chemicals for evaluation; and chemicals for special applications.

## Sensory Panels

● Foster D. Snell, Inc. announces the availability of a new booklet describing the uses of sensory panels. Sensory panels are valuable in the evaluation of foods and beverages for odor and flavor, and for aid in formulation. Panels are useful in flavor research, the evaluation of packaging material, and in the investigation of off-odors and flavors. Copies of the booklet may be obtained from Foster D. Snell, Inc. 29 West 15th Street, New York 11, N. Y.

## Hydroxyethyl Cellulose

● A new 16-page booklet, describing the properties and use of Cellosize hydroxyethyl cellulose, has been published by Union Carbide Chemicals Company, Division of Union Carbide Corporation.

The booklet contains extensive data on properties, preparation of solutions, film and coating properties, preparation of water-insoluble films, applications, toxicological properties, specification limits, shipping data and test methods.

## PVP/VA Copolymers Bulletin

● An expanded and comprehensive bulletin on new types of commercially available vinylpyrrolidone/vinyl acetate copolymers and their numerous applications has just been issued by Antara Chemicals, sales division of General Aniline and Film Corporation. These materials were first introduced as 50 per cent ethanol solutions. They are now available in various physical forms as well as in several monomer ratios. Solid, emulsion and solution types now provide specific materials suitable for diverse processing conditions and product requirements, and for use in aqueous as well as organic solvent or aerosol systems. This two-color, 20-page publication outlines recently developed uses of these acetylene-derived chemicals. Technical data are presented in charts, and practical formulations are given for use in adhesives, hair-grooming agents, protective films, shoe polish, plant spray, decorative sprays, and anti-tarnish coatings.

## Pharmaceutical Problems

● A new 4-page folder, titled "X-ray Analysis of Pharmaceuticals," is available from Philips Electronics Instruments, 750 South Fulton Avenue, Mount Vernon, New York. Illustrated with photos, the text describes work with X-rays reported by Parke-Davis & Company in solving many problems. Investigations involve zinc content of rubber closures, iron content of clays, selenium in steroid preparations, merthiolate in protein mixtures, organic bromine in cough syrup, hyoscyne in motion sickness medicine, hydrogenation and high pressure reactions on catalysts, iron oxide in vitamins, and inert materials in antibiotics.

## Westfalia Bulletin

● An eight-page bulletin describing a series of WESTFALIA Centrifugal Separators especially designed for the purification and dehydration of oils and fats has just been released by Centrico, Inc., Englewood, N. J. Printed in four colors, the new bulletin features detailed descriptions and cut-away drawings of WESTFALIA type OM, OLM, OSM and OSL Separators.

The series of centrifugal separators described in the new bulletin are useful in the polishing and de-watering of vegetable and animal oils and fats containing relatively small amounts of solids—such as palm oil, olive oil, fish oil, butter oil, lard and tallow.



# News

## and Events

### D&O HOSPITALITY AT C.S.M.A. CONVENTION



This early evening photo taken in the Dodge & Olcott Hospitality Suite during the C.S.M.A. Convention includes, from left to right, Mat Phelan, D&O Philadelphia branch manager and sales representative; Dr. Robert F. Prindle, Lehn & Fink Products; William D. Williams, National Labs, Inc.; Herb Kainik, D&O Technical Director, Perfume Sales; Fred G. Taylor, Lehn & Fink Products; H. L. Melzer, National Labs, Inc.

### Robert A. Kramer Visits South America

Robert A. Kramer, vice president in charge of sales for Evans Chemetics, Inc., journeyed last month to South America for a three-week business trip. During the trip he visited various scientific laboratories, chemical plants and governmental agencies of Peru, Brazil, Chile and Argentina. Mr. Kramer, who is also secretary of the Society of Cosmetic Chemists, spoke to groups in various countries in an effort to interest them in the newly formed International Federation of Societies of Cosmetic Chemists.

### Pennsalt Chemicals to Build New Plant

Pennsalt Chemicals Corporation will invest about \$500,000 in a new blending, packaging and warehousing plant at Atlanta, Georgia. Scheduled to go on stream in the Spring of 1960, the Atlanta plant will be the seventh unit devoted to production of Pennsalt specialties added in the past five years.

### Evans Research Names Three Project Leaders

Ralph J. Addesso, Frank Ellison, and Max Marti, have been appointed Project Leaders at Evans Research and Development Corporation, New York City.

Ralph J. Addesso, Project Leader in analytical chemistry, was formerly with United Testing Company, Inc. and Fleischmann Laboratories of Standard Brands, Inc. in the capacity of Senior Analytical Research Chemist. He joined Evans Research in 1957.

Frank Ellison, Project Leader in the chromatography section, has been with Evans Research since 1951. During this time, he has applied paper and gas chromatography to help solve numerous diversified research problems.

Max Marti, Project Leader in the field of product development, was formerly Technical Director of Salea, A.G., Zurich, Switzerland where he specialized in hair preparations and obtained several patents on his inventions. He joined Evans Research in 1957.

### Givaudan-Delawanna Holds Annual Christmas Party

Approximately three hundred and forty employees attended the annual Christmas Party of Givaudan-Delawanna, Inc., and were treated to an enjoyable evening of song, dance and refreshment, including a traditionally excellent steak dinner. The party was held at the Swiss Chalet, Rochelle Park, New Jersey, on Saturday, December 12, 1959. Six employees who had completed twenty-five years of service during 1959 were honored during the party and were presented with Gold Swiss watches.

The holiday-spirited gathering was briefly addressed by Mr. E. R. Durrer, President, and Dr. Max Luthy, Vice President in Charge of Production and Research, reflecting on the progress made by the company during the ten years that have elapsed since its own silver anniversary in 1949.

### 25-YEAR EMPLOYEES HONORED BY GIVAUDAN



At the presentation of gold Swiss watches to Givaudan's new 25-year employees (l. to r.) Arend Valder Valk; Joseph Miskiv; Rudy Angelino; Ruth Uhlig Sheridan; Dr. Max Luthy, vice president in charge of production and research; Mr. E. R. Durrer, president, making the presentation; Urho Korpi; and Fred Jaeckel.



## Daniel Van Dyk Elected Hazel Bishop President

The Board of Directors of Hazel Bishop, Inc., recently elected Daniel Van Dyk president and chief executive officer of the corporation, it was announced by Raymond Spector, chairman.

An amount of \$1,400,000 in cash was paid into the treasury by Mr. Van Dyk and his associates for the purchase of 350,000 shares of the company's common stock.

It was further disclosed that Mr. Van Dyk and his associates have obtained an option from Mr. Spector to purchase 300,000 shares of Spector's personal stock at \$6.00 per share. Spector stated that in addition he has assigned to Mr. Van Dyk all voting rights to the 325,000 shares he will still own after the anticipated exercise of the option.

Well-known in marketing circles and a leading industrialist and financier, Mr. Van Dyk is a former president of Ever-sharp Incorporated and a former merchandise counsellor for R. H. Macy & Co. He is also a director of General Acceptance Corporation, Eversharp Incorporated, Clinton Engines Corporation and the Stuyvesant Insurance Company.

Although retaining his directorate in



these companies, Mr. Van Dyk revealed plans to devote his energies to the presidency of Hazel Bishop, Inc. He said a new aggressive sales, advertising and promotional campaign is planned, as well as a broadening and diversification of the company's product line, which includes lipsticks, eye makeup, nail polish, hair-spray and other beauty preparations.

The Board also announced the election of a five-man executive committee to be

headed by David B. Charnay as chairman.

The new nine-man Board of Directors will consist of Mr. Spector, chairman; Mr. Van Dyk, president;

Mr. Robert Bragarnick, formerly vice president and director of marketing, Revlon, Inc., and of Seagram, Inc.;

Mr. David B. Charnay, chairman of the board, Highway Trailer Industries, Inc., and Clinton Engines Corporation;

Mr. Julius Epstein, director numerous real estate and hotel corporations, also of Spector Mid-States;

Mr. Herman L. Johnson, Jr., executive vice president, Hazel Bishop, Inc.;

Dr. Charles McKhann, director, Stanley Warner Corporation (Playtex), American News and Chemway;

Mr. Albert Parker, senior partner, legal firm of Parker, Chapin & Flattau, and director, Inter State Department Stores, director, Mead & Montague, Inc.;

Mr. Wickliffe Shreve, partner, Hayden, Stone & Co.; members, New York Stock Exchange, also director of Air Express International Agency, Bankers Federal Savings & Loan, Knott Hotels, General Transistor, and Cohu Electronics.

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CARMEL MYERS HOSTS COSMETIC SALES PERSONNEL




Carmel Myers (standing third from the left) sole distributor of Fragonard's perfumes, Zizanie and Gamin, entertained a group of the Cosmetic Sales Staff from the New York stores at a pre-Christmas party at her penthouse apartment at the Madison Hotel.

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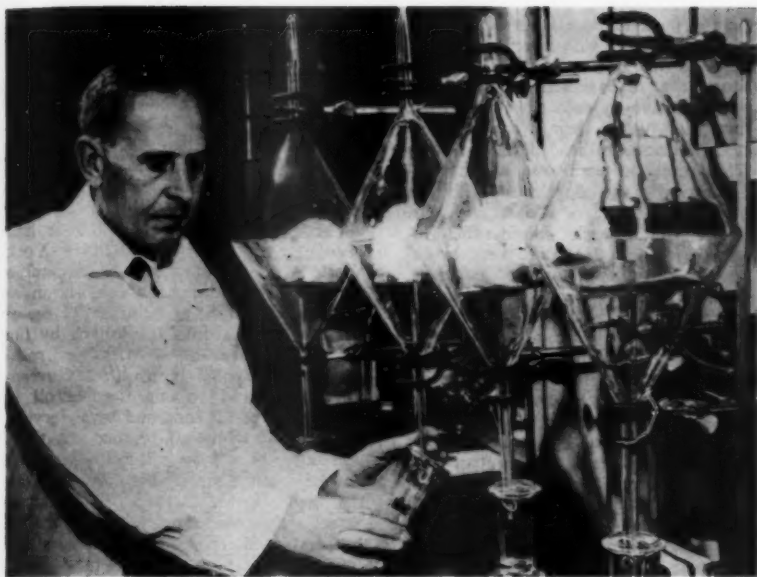
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## The Toni Company Dedicates Expanded Research Laboratories

The Toni Company has officially dedicated its expanded research laboratories in the Merchandise Mart, Chicago, largest in the world devoted to the study of cosmetic products for use on hair and skin.

The newly-expanded research laboratories are located on the fourth and fifth floors of Chicago's sprawling Merchandise Mart and are adjacent to the company's executive offices.

The laboratories are completely air-conditioned and equipped with the most complete modern laboratory equipment. Among the special units are: a Shirley Ferranti viscometer, a high speed digital computer, polargraphic equipment, an infrared spectrometer, constant temperature rooms and a wide range of special and standard fiber testing instruments.

Most of Toni's research work is carried out in the Chicago laboratories, where studies are directed toward the physical and chemical study of hair and skin and the effects on them by various products and processes. New chemicals are synthesized; experimental formulations are prepared and tested; competitive products and methods are evaluated.

A large beauty clinic also is maintained in the Merchandise Mart, where experimental waves and shampoos and other tests are conducted on thousands of women volunteers every year.

The research staff of about 200 scientists and technicians is headed by Raymond E. Reed, vice-president in charge of technical operations, and Dr. Ray C. Houtz, director of research. The largest



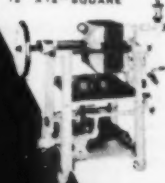
in number are chemists, specializing in organic, inorganic, physical, analytical and biochemistry. Others on the staff include histologists, biochemists, microscopists, physicians, statisticians and dermatologists.

The objectives of this group are to insure medical, chemical and physical safety and efficacy of products; to make these products esthetically attractive and pleasing; to maintain quality and product uniformity; to improve present products and to develop new products and processes. The scope of their research activities includes toxicological studies on animals, dermatological patch tests on humans, off-the-head tests on human hair, testing under practical use conditions with live models.

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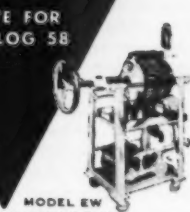
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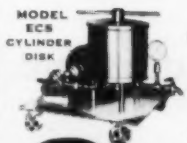
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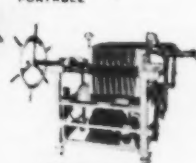
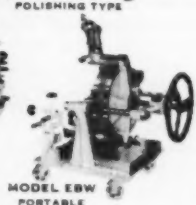
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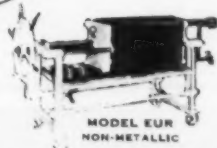
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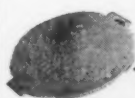
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### Firmenich Inaugurates New Research Laboratory

On the 23rd October last, Firmenich & Co., Geneva (Switzerland) officially inaugurated their new Research Laboratories, the construction of which has taken place over the last four years.

The ceremony was opened by Dr. Roger Firmenich, one of the partners of the firm, who welcomed the numerous representatives of the Swiss Federal authorities, the Canton of Geneva and the university organizations. In all, more than 120 people were present.

In his opening speech, Dr. Roger Firmenich emphasized the evergrowing importance of scientific research without which the creation of new products in the perfumery and flavour fields would today be practically impossible. The increasing demand, especially for flavoring essences, and the high cost of certain natural products, render research work indispensable, in the first place for the creation of new methods that will permit a reduction in the cost of those syntheses previously realized. As an example the speaker mentioned that natural vanilline at present would cost something like Sw.fr.5000.-per kilo whereas synthetic vanilline sells at only Sw.fr.25.-per kilo. Moreover, the world consumption of vanilline could never be covered without the synthetic variety. It is for these reasons that Firmenich & Co. have placed at the disposal of their research depart-

ment the most modern laboratories conceivable equipped with precision instruments of the latest design. In this manner the management of the firm have been able to demonstrate to what extent they value their research team and in particular its head, Dr. Max Stoll.

This research team was not created spontaneously, but developed with the years. The founder of the factory, Dr. Philippe Chuit, took considerable interest in research from the very beginning. In fact, he and his associates, Martin Naef and Frederic Firmenich, were amongst the first in Switzerland who, immediately after the first world war, ventured to undertake real scientific research with the collaboration of Professor Ruzicka, holder of the 1939 Nobel prize in chemistry, then lecturer at the Swiss Federal Polytechnic School in Zurich. This permanent contact with a university organization led to the creation of the research team and had a beneficial influence on its methods and conception of work. These connections with academic research have been maintained and developed, three new centers having in fact been created: one in 1947 in Paris, the other two in 1955 and 1958 respectively in Boston USA.

Dr. Roger Firmenich concluded with a description of the equipment contained in the new building, as explained in detail in the illustrated booklet that has been sent to the customers of the firm throughout the world.

### Mel Pollack Joins Fritzsche Quarter-Century Club

Mel Pollack of Fritzsche Brothers' Export Division is the latest member of that organization to be admitted into its exclusive and distinguished Quarter of a Century Club. He becomes the sixth employee to be so honored during 1959. Prior to joining the New York essential oil and chemical firm, Mr. Pollack had been employed by the old "New York World", V. Vivaudou, and E. R. Squibb & Company. A luncheon celebration at Charles Restaurant on Thursday, December 17th, was given Mr. Pollack by the officers and directors of Fritzsche Brothers, who presented him with a Government bond and an engrossed scroll in recognition of his long and loyal service. Responding with appreciation to these and other gifts from his fellow employees, he recalled vividly his first year with the firm—then located at 78 Beekman Street—when an early morning fire all but destroyed its plant, laboratories and offices. For months afterwards, Mel's papers and files were permeated with the assorted "fragrances" to which they had been subjected on that catastrophic day. Marking a turning point in the Company's history, this proved—in effect—to be a fortunate "calamity," for it precipitated Fritzsche's subsequent move to the then new Port Authority Building and to the vastly expanded facilities it now occupies.

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George McAteer



Jack Jecker

#### Kartridg-Pak Appoints Two Representatives

Kartridg-Pak Co. has announced the appointment of two new sales and service representatives in the Atlantic coast region. George McAteer, for eight years Eastern Representative with the company's Davenport, Iowa division, has been named Eastern Sales and Service Representative covering New York, New England, Central and Eastern Canada. At the same time, Jack Jecker, formerly Eastern Service Representative, has been named Southeastern Sales and Service Representative. Jecker has been with The Kartridg-Pak Company and Mojonnier Associates for seven years.

## Redbook Beauty Survey Shows Younger Women are the First to Try New Beauty Products

Age is a distinct factor in a woman's attitude toward trying new beauty products, according to a survey released today by Redbook Magazine.

The 75-page study, entitled "Redbook Magazine's 1959 Beauty Survey," covers the daily beauty habits and routines of some 5,512 women. It delves into the use of basic products, new products tried, shopping habits and expenditures for beauty aids. Among the specific items covered are nail polish and enamel, lipstick, creams and lotions, head coloring, permanents, shampoos, deodorants, reducing machines and systems.

The survey shows that as a woman becomes older, she is less likely to experiment with new cosmetic products. A 25.5% larger proportion of the younger women said they tried new beauty and cosmetic products in the last year than did older women. The younger women also tried a wider range of new products. Topping the list of new products tried by older women was face creams. Among the younger women, lipstick topped the list.

There is also a wide difference, the survey states, in the expenditures for cosmetic and toiletry products by age group. The median expenditure for all women interviewed in the Redbook survey was \$32.52 per year. Women in the 18 to 34 age group spent the most—\$34.80 a year—47.2% more than the amount spent by women in the oldest group.

Younger women also apply fresh make-up more often than older women. 68% of the women 18 to 34 put on fresh make-up two or more times a day—com-

pared to 63% of women 35 to 49, and only 54% of the women 50 and over.

The routine followed by women before putting on new applications of make-up varies by age group. Over half of the younger women "wash" while older women apply creams and liquid cleansers.

Among the married women interviewed, more of the younger women "cleanse their face" and "apply fresh make-up" before their husbands come home than do older women.

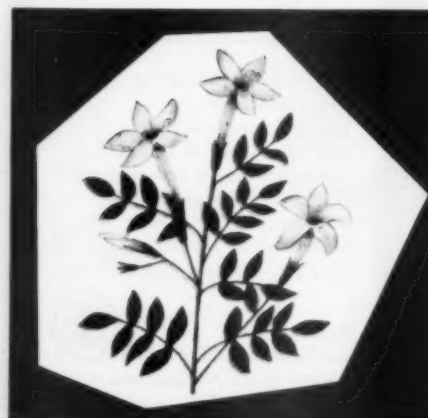
The study shows a woman's age may also determine whether she buys her cosmetics in a drug store, department store or supermarket.

In the downtown shopping area, the study shows department stores were the number one place to purchase cosmetics among older women. Younger women gave more business to supermarkets than older women; a little more to variety stores.

Within the shopping center areas, the study shows drug stores are clearly ahead as the prime outlet with all age groups. The younger women rank supermarkets as their second choice, department stores third. Among the older women the pattern is reversed. A greater percentage of older women buy in department stores in the shopping centers than they do in supermarkets.

In neighborhood shopping areas, four out of five women in all age groups bought in drug stores. Supermarkets are in second position. Younger women show heavier use of supermarkets.

This Redbook Magazine beauty survey was based on 5,512 mail questionnaire interviews with Redbook subscribers.



Esperis s.a.





Polak's Frutal Works, Inc., manufacturers of perfume and flavoring raw materials, held their Annual Sales Meeting December 14 thru 16 at their plant in Middletown, N.Y. A dinner party was held at the Orange County Gold Club on December 15 for the Executive, Sales and Technical Staff and their ladies. In the above photo, standing left to right: E. Johnson, D. Mancino, P. Delaney, L. Pais, E. Sturman, J. Donnelly, K. Freiberg, A. Krol, A. J. de Jong, Dr. J. S. Jellinek, M. Preiser, F. Marshall, H. Lonergan, E. Buday, J. Melody. Seated left to right: E. Vles, Dr. R. Boucher, A. H. Micheels, E. Polak, W. R. Lloyd (Ass't Managing Director of Soffor, Ltd., England), B. Polak, president, J. Meihuizen (Co-Director of P.F.W., Holland), J. ter Veer, L. Kirsh, H. Roger (Far Eastern representative).

#### CORRECTION

In announcing the names of the members of the "Working Committee" for the INTERNATIONAL FEDERATION OF SOCIETIES OF COSMETIC CHEMISTS (October issue) Dr. P. Velon was erroneously listed as a member. Mr. Kurt Pfeiffer in fact is the third member; however, Dr. Velon was a delegate from France. We regret this transposition in names.

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## Hazleton Laboratories Completes New Building

Hazleton Laboratories, independent biological research firm, has just completed construction of a new building at its headquarters in Falls Church, Virginia. The new building, the first of several scheduled in a long-range expansion program, will add 10,000 square feet of research facilities for studies on the effects of chemicals on biological systems.

Construction on another 12,000-square-foot building is scheduled for next spring. While designed for eventual conversion into a research laboratory, this will serve as a new administration building.

## Naarden's Interests Extended to Spain

The N. V. Chemische Fabriek "Naarden" has announced an extension of its activities abroad by the addition of an outlet in Spain under the title "Naarden" Productos Orgánicos S.A. in Barcelona. The new factory, which is now under construction, will shortly commence production of basic materials for the food-stuffs industries. These will include bulk products such as sodium benzoate and benzoic acid.

## PCPS Announces Faculty Title Changes

Five members of the Faculty of the Philadelphia College of Pharmacy and Science have been advanced in title fol-

## NOTABLES AT GIVAUDAN RECEPTION



Enjoying a laugh at the Givaudan cocktail party are (left to right) Mr. Andre Wick of Heubigant; Mr. H. Gregory Thomas of Chanel; Givaudan's President, E. R. Durrer; Mr. J. Manoha of Heubigant; Pierre Bouillette of Givaudan and Mr. S. Koss of Heubigant.

lowing the confirmatory action taken at a recent meeting of the Board of Trustees. These new titles, according to an announcement by Dr. Ivor Griffith, President of the College, are: Professor Robert E. Abrams, Professor of Pharmacy Administration; Dr. Martin Barr, Professor of Physical Pharmacy and Pharmaceutical Research; Dr. Grafton D. Chase, Director of the Radiochemical Laboratories; Dr. Alfonso R. Gennaro,

Associate Professor of Chemistry; and Dr. Nathan Rubin, Professor of Organic Chemistry.

## Jean Warner Baer Joins Sprapak Chemicals, Inc.

Jean Warner Baer, Aerosol Consultant has been appointed managing and technical director of Sprapak Chemicals, Inc., a new aerosol packaging company.



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### Antara Announces Field Sales Shifts

The following appointments and changes affecting Antara Chemicals' Field Sales Force were announced today by Thomas R. Moore, Field Sales Manager, Antara Chemicals, a sales division of Dyestuff and Chemical Division, General Aniline & Film Corporation:

Alfred W. Purdy, until now a sales engineer in the company's Philadelphia office, is being promoted to Eastern Regional Manager, in which position he will be located in the company's home office in New York. Mr. Purdy joined General Dyestuff Company in New York in 1935, then moved to Antara Chemicals as a chemical salesman for the Philadelphia area. In 1947 he became a sales engineer for the company in Philadelphia, a position which he held until his current advancement.

John P. Miller replaces Mr. Purdy as a sales engineer for the Philadelphia area. Mr. Miller joined General Aniline & Film Corporation at the Central Research Laboratories in Easton, Pennsylvania, in the position of technical service chemist. He then transferred to Antara Chemicals in 1952, as an application research chemist, moved to the marketing department in New York in 1954 as a sales correspondent, and then to St. Louis in 1958, to become a sales engineer.

Joseph A. Czerwinski is being moved into Mr. Miller's previous position of

sales engineer in the St. Louis area.

Two additional changes concern William C. Brunson and Eugene M. Kennedy. Mr. Brunson is being transferred from Atlanta, Georgia to the company's New Jersey territory. Replacing Mr. Brunson in Atlanta, Mr. Kennedy has been appointed a sales engineer for that area.

### Smith Kline & French Releases Medical Film

Smith Kline & French Laboratories this week announced the release of a new medical film, "The Neurologic Actions of Phenothiazine Compounds," for special professional audiences. The 30-minute, color presentation is designed as a discussion film for staff psychiatrists, physicians and nurses in mental hospitals as well as for the neuro-psychiatric staffs in general hospitals.

Jack Borland, director of SK&F's Medical Film Center, said the film outlines the anatomy and the functional relationships of the principal central nervous system structures thought to be involved in the action of the phenothiazine compounds.

Included in the film which features both animation and live action scenes, are electroencephalographic tracings from both cortical and subcortical regions of monkey brains. In addition, neurological studies in animals which have provided clues as to sites and mechanisms of the actions of the phenothiazine compounds

also will be demonstrated. The chemistry and clinical observations regarding the area of phenothiazine activity also are reviewed in the film.

Produced for SK&F by Ansel Film Studios, Inc., the 16 mm. film is presently available on a free loan basis only to professional personnel in the psychiatric field.

### Heyden Newport Building Maleic Anhydride Plant

Heyden Newport Chemical Corporation is proceeding with the building of its new maleic anhydride plant at Fords, New Jersey. The new plant, the first maleic anhydride venture for Heyden Newport, is being designed and built by Scientific Design Company, Inc. to use the Scientific Design process and will have a capacity of 24,000,000 pounds per year. The anticipated onstream date for the new plant is during the fourth quarter of 1960.

### OBITUARY

#### Michael J. Coppola

Michael J. Coppola, widely known in the aromatics and aerosol trades as a sales representative of the Du Pont Co. for the last 40 years, died of a heart attack while attending the 46th annual meeting of the Chemical Specialties Manufacturer's Assn. in Washington, D. C., on the evening of December 8.

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## PERSONALITIES



R. E. Eckton

**R. E. Eckton**, Technical Service Manager of Givaudan and Co., Ltd., Whyteleaf, Surrey, England, has just completed a three week visit to the United States. During his stay, Mr. Eckton toured the Givaudan facilities here and studied market conditions in America. He reviewed new aromatic chemical and perfume oil development and their application in the British market.

**Abraham Seldner** has been appointed director of the Special Services Department of Albert Verley & Co. He will assist customers with the formulations of cosmetics, aerosols, household specialties, as well as application of all types of Verley products.

**Graydon Cass** has joined Thomasson of Pa., Inc., custom packagers of aerosol products, as supervisor of the Thomasson laboratory staff, in charge of quality control and product development. Mr. Cass joins Thomasson with 14 years of aerosol development experience at Crown Cork & Seal Co., where he was in charge of Crown's extensive aerosol laboratory activities.

**Neil H. McElroy** was elected chairman of the board of The Procter & Gamble Co. recently. Mr. McElroy was president of Procter & Gamble from 1948 to 1957, when he resigned to become Secretary of Defense. He was succeeded as company president by Howard Morgens, who remains in that position. It was announced that the chairman of the board and the president will share executive responsibilities in the way they consider to be in the best interests of the company. Mr. McElroy joined Procter & Gamble in 1925. He began in the mail room and advanced through various positions to become president 23 years later.



Dr. William A. Meer

**Dr. William A. Meer** has been appointed director of the Quality Control Laboratories of the Meer Corp. Dr. Meer will supervise all steps of the company's quality control program from collection in the field through final processing of botanicals and drug and flavor extracts.



David X. Klein

**David X. Klein**, technical director of Heyden Chemical Division, Heyden Newport Chemical Corp., has been appointed a divisional Vice President, according to a recent announcement by Mr. S. Askin, the corporation's president. Mr. Klein, technical director for the recently formed division since June 1959, has been responsible for the division's expansion and new products program. He was formerly manager of research at the New Jersey laboratories.

**Ed Frindel** has joined Sofskin, Inc., as merchandise manager, according to a recent announcement by Alfred Roberts, president. Mr. Frindel was formerly Field Sales Manager for 5-Day Laboratories and he was also Divisional Sales Manager for the Barbasol Co.



Dr. Ernst T. Theimer

**Dr. Ernst T. Theimer**, director of research for International Flavors & Fragrances Inc., was selected Chairman-Elect of the North Jersey Section of the American Chemical Society at its recent election. The North Jersey Section is the second largest of 155 regional sections of the American Chemical Society.

**Roger M. Kirk, Jr.** has been named sales manager of the Lehn & Fink Division of Lehn & Fink Products Corp. He replaces Robert W. Naething, who resigned December 31. In his new post, Mr. Kirk will direct field sales and participate in the division's marketing management team.

**Kevin J. Solon**, assistant to the vice president and general sales manager of the Glass Container Division of Owens-Illinois, has been appointed director of product planning and marketing research for the company's Paper Products Division.

**Joseph DiSomma** has been appointed as a chemist in the Oral Products Section of the Research and Development Dept. of Colgate-Palmolive Company's Toilet Articles Division, according to a recent announcement by Dr. Kenneth L. Russell, Director of Research and Development for the Department.

**Milton Gold** has been advanced to the position of Controller of Warner-Lambert Pharmaceutical Co., according to a recent announcement by Alfred E. Driscoll, president. Since 1957, when he joined the company, Mr. Gold has been Manager of Audits, Systems and Procedures.



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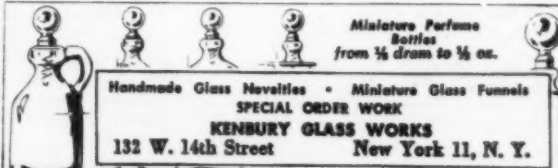


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A DIVISION OF VERONA-PARMA CHEMICAL CORP.

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